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SPONSORED PROJECT INITIATION

Date: 3/28/80

Project Title: Reactive Sputtering of Tin and Solid Lubricants and Shear Strength Testing of Solid Lubricants

Project No: E-25-630

Project Director: Dr. Ward Winer

Sponsor: Hughes Aricraft Company

Agreement Period: From 2/6/80 Until ~~10/15/80~~ 12/31/80

Type Agreement: Purchase Order No. 04-406163-FS5

Amount: $59,932 (fixed price)

Reports Required: Monthly Progress & Financial Reports, Basic Program Schedule, Comprehensive Reports, Final Report

Sponsor Contact Person (s):

Technical Matters

Contractual Matters
(thru OCA)

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Defense Priority Rating: DO-C9 under DMS Reg 1

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SPONSORED PROJECT TERMINATION SHEET

Date 6/15/82

Project Title: Reactive Sputtering of Tin and Solid Lubricants and Shear Strength Testing of Solid Lubricants

Project No: E-25-630

Project Director: Dr. Ward Winer

Sponsor: Hughes Aircraft Company

Effective Termination Date: 12/31/80

Clearance of Accounting Charges: 12/31/80

Grant/Contract Closeout Actions Remaining:

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- [ ] Final Fiscal Report
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FORM OCA 10:781

E25-630(64?)

PROGRESS REPORT

Reactive Sputtering of TiN and Solid Lubricants and Shear Strength Testing of Solid Lubricants

CONTRACT NO: 04-445624-SLI

SUBMITTED TO:

M. N. Gardos
Program Manager
Technology Support
Hughes Aircraft Company, M.S. D-133
Culver City, California 90230

SUBMITTED BY:

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Principal Investigators
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16 September 1981

I. Hard Coating - Process Development

In the first phase of this program it was shown that non-distortive hard coating techniques can be used to deposit a thin layer of titanium nitride on the working surfaces of advanced bearings. Magnetron sputtering technique was shown to be suitable to deposit 1 to 5 μm thick layers of titanium nitride. The coating system operated in the DC reactive mode allowed the deposition of golden-yellow titanium nitride on hardened AISI 52100 test sample surfaces. The test program called for the evaluation of friction and film adhesion characteristics on Faville-6 samples at 50 psi nominal contact stress at 900 rpm. Thrust bearing tests at Hertz stresses of about 160 ksi were also called for.

The principal thrust of the first phase activity was to demonstrate that the requirements mentioned can be met successfully with magnetron reactive sputtering. The requirements were met by the use of proprietary surface conditioning agents (2% solution of a chemical package marketed by W. T. Bean Company of Detroit, Michigan) and by the establishment of optimal surface preparation procedures. This alleviated the coating adhesion problem which until then prevented the non-distortive hard coating of hardened steel surfaces (Refer to Rigert's earlier work on reactive sputtering of TiC on hardened steel surfaces).

Figures 1a through 1d show the appearance of the TiN-coated virgin and tested Faville-6 sample surfaces. Thrust bearing surfaces prepared with the Bean chemical package exhibited similar small scale pitting as a result of localized chemical attack. Small scale de-bonding occurred in the vicinity of the pits, Figure 1c, and the presence of reaction products prevented effective local film adhesion. Despite this difficulty, the coated Faville-6 samples and the thrust bearings met all test requirements.

Phase II of the advanced bearing program requires hard coating of entirely new materials (M-50 and T-15 steels; hot pressed silicon nitride). The pertinent test programs call for much higher contact stresses both in Faville-6 tests (up to 4000 psi at 4000 rpm) and in thrust bearings (Hertz stress to 260 ksi) at elevated temperatures (600°F). The cleaning and coating procedures developed for hardened AISI 52100 steel are not entirely applicable for the new materials. The more severe test conditions do not allow the presence of pitting artifacts. Hence the first part of the present program was devoted to establishing cleaning procedures and coating extensive coating trials to assure non-distortive coating without pitting artifacts.

Figures 2a and 2b show that the need to produce adequately bonded films of hard coatings can be met with the use of Oakite HD 126 for surface preparation. The details of the surface preparation procedure are described in Hughes Standard HP 7-31, pages 3 to 6 (Restricted usage document made available by Mr. M. N. Gardos). Following surface cleaning as outlined in the cited Hughes document, the test samples are placed in the coating system and pumped down to a pressure lower than 0.8×10^{-6} Torr. While at this pressure, the samples are radiant heated to a temperature sufficient to desorb water vapor in 30 minutes. This temperature, monitored with a thermocouple, ranges between 400^{o}F and 700^{o}F (Note: To prevent loss of temper, low alloy steels such as AISI 52100 cannot be heated above 400^{o}F. The M-50 and T-15 steels are tempered in the 1000^{o}F to 1150^{o}F range and allow coating following heating to 800^{o}F without detriment). Back sputtering following heating is an assist to produce coatings that do not debond (It has so far been found to be not necessary.) Test samples are then sputtered under conditions identified in the first phase of the advanced bearing program.

Before the Hughes standard HP 7-31 became available, to eliminate pitting and to produce well-bonded TiN coatings, an extensive RF reactive magnetron sputtering test program was undertaken. A new RF power supply (1.5 kw, Plasmaloc-2 system, 25 to 125 KHZ) was procured and installed without disturbing the D.C. magnetron coating system in any manner. Following a number of trials, it became possible to RF sputter at pressures of 1 to 2 x 10^{-3} Torr (low pressures assure low propensity for argon occlusion in the film deposited). Hard coatings are produced at a gas composition of 30% Nitrogen when target power density is between 40 and 50 watts/cm^2. In TiN coating with RF sputtering, substrate heating is not necessary even when AISI 52100 steel samples are coated. Use of a rotary fixture to rotate test samples during coating is a significant assistance for coating adhesion and uniformity.

II. Hard Coating - Test Samples Coated

The improved coating procedures (D.C. Magnetron and RF Sputtering) that were developed were used to coat inner races of test Air Research ball bearings furnished by Mr. M. N. Gardos. Using a rotary fixture, the bearings were coated at a target to sample distance of 6 cm. The coated samples, one DC magnetron coated and the other RF sputtered, have been furnished to Mr. M. N. Gardos for test purposes.

A total of 12 test samples, bearing inner races, have been received from Battelle-Columbus for TiN coating. The batch contained AISI 52100 and M-50 inner races commercially finished. Four of these have been coated (D.C. Magnetron and RF Sputtering) and made available to Mr. M. N. Gardos for transmittal to Mr. J. Kannel at Battelle.

At Mr. Kannel's request, the remainder of the test samples are being held (to be coated following initial evaluation).

A large batch (50 sets each) of M-50 and T-15 Faville-6 test samples and 52100 thrust bearings have been received from Hughes. They are to be TiN coated and tested (at room temperature and at 600^{o}F) at Georgia Tech. Some are to be furnished to an overseas contractor for turbo-lube coating. A specially constructed rotary coating rig has been constructed for the simultaneous coating of three *sets* of Faville-6 test samples at a time. Initial trials have been conducted successfully and the rig is ready for routine usage.

Earlier studies of RF Sputtering of MoS_2 demonstrated a preferred texture unique to relative orientation. Coating under normal incidence conditions appeared desirable. A ball coating fixture allowing coating under normal incidence conditions was constructed and tested. Ball masks were used in a rotary fixture allowing random motion.

The masking does not allow back sputtering of balls prior to coating. Under magnetron coating conditions, the coating flux is large. Fixture contamination is extensive with difficult housekeeping problems. The rotary coating fixture for balls is not considered to be suitable. A new ball coating device is now under construction. It is expected to be operational by the end of October 1981

(The device is similar to that being used at Hughes for MoS_2 coating of vacuum bearings).

III. Coating Evaluation Rig - 600°F Tests

The existing traction rig cannot be used to measure the traction of coated test samples at 600°F. A new rig has been designed and nearly completed. A schematic of the cross section of the device is shown in Figure 3. This device is suitable for tribo-testing of coated test discs (M-50; T-15 and HPSN). The same rig is to be used for sliding friction and life tests, rolling friction and life tests, and for variable slide-roll traction measurement.

In traction measurement under variable slide-roll ratio condition, the rig will permit slide-roll ratio in the +6% to -6% range. The contact geometry is that of a crowned roller against a disk. Contact load is accomplished pneumatically by a piston acting against the lower disk spindle bearing. Both the disk and the roller are driven through stainless steel chains and sprockets by a single motor. Traction will be measured at the upper disk spindle support bearing.

The high temperature will be maintained by resistive heating in a Macor box. The box has been operated at 400C (Figure 5) with all internal parts in place. Both the disk spindle and the roller spindle are hollow to reduce conductive losses from the Macor box.

This machine will also be capable of performing the Falex 6 type of sliding and rolling test. See inserts in Figure 3. Friction is then measured by a torque arm and load cell affixed to the support spool for the lower test piece.

IV. Modified High Load Friction and Life Tester

To facilitate the rapid evaluation of coated specimens at high loads, a high temperature friction and life tester has been constructed (Figures 6 and 7). Sliding or rolling Falex 6 type specimens are pneumatically loaded in an oven consisting of a resistively heated, fired lava box. Normal force and frictional torque are measured by a two component transducer. The upper specimen is fixed to and driven by the spindle of a drill press. All mechanical parts are hollow with a small section area where they pass through the lava box to reduce conductive losses.

This system is a modified version of the test rig used in the first phase of the advanced bearing program. Modifications are made necessary by the rather extensive test program (nearly 200 tests) requested by Mr. M. N. Gardos. The revised tests also require more severe tests (4000 psi contact stress at 4000 rpm in Faville-6 tests;

Hertz stresses to 260 ksi in thrust bearings at 4000 rpm). The modified high load tester is expected to be satisfactory for loads up to 500 lbs (2500 psi in Faville-6 samples) at $600^{\circ}F$. Although the drive is capable of 4000 rpm, the upper usable limit remains to be found. The upper load also remains to be determined (chatter problems may occur).

Details of system capability will become evident in October and will be summarized in the progress report for October.

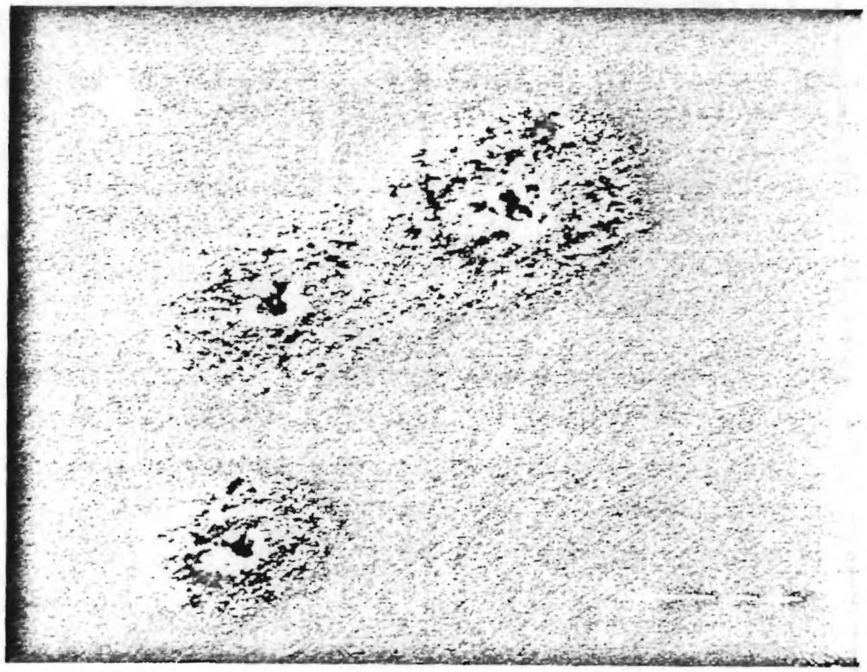

Figure 1.a. Virgin surface of TiN coated Faville-6. Test sample of hardened AISI 52100 steel. Note surface contamination at pit. Magnification 690X.

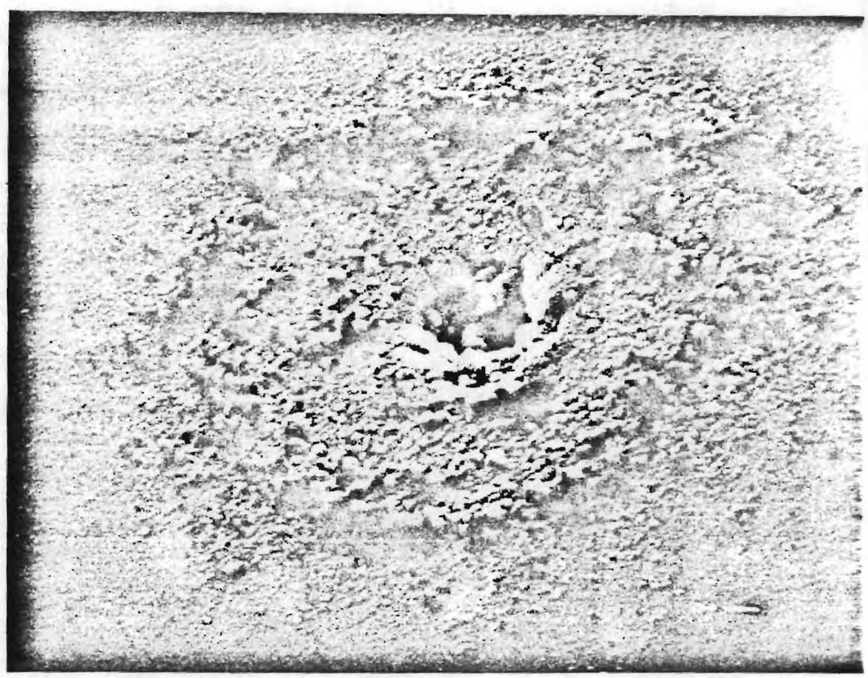

Figure 1.b. Same as above at 1500X. Reaction products at pit locations produces poorly bonded TiN in the vicinity of pits.

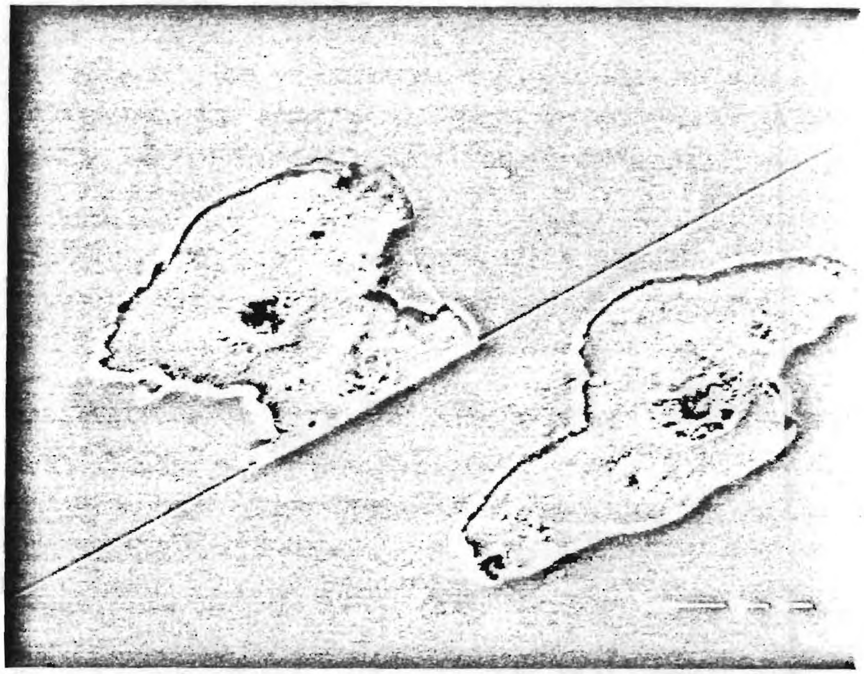

Figure 1.c. TiN coated surface of AISI 52100 hardened steel. Faville-6 test sample. Note pitting and small scale debonding on coated test sample surface following testing. Magnification 690X.

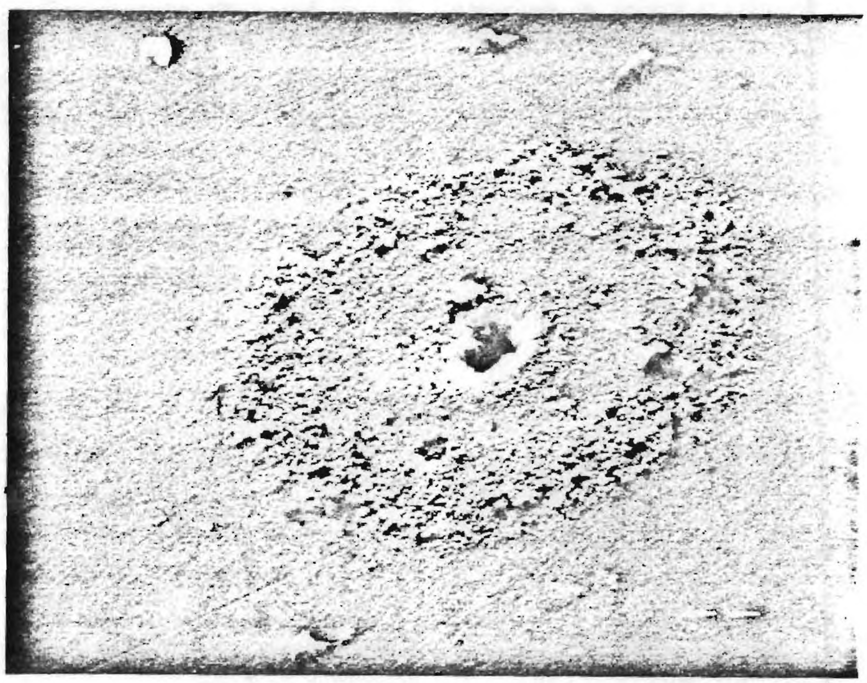

Figure 1.d. Appearance of a WSe_2/In/Ga alloy coated test sample initially coated with titanium nitride. Hardened AISI 52100 steel substrate. Surface prepared with Bean's Metal conditioner. Note surface contamination and absence of soft coat at pit locations. Magnification 2200X.

Figure 2.a. TiN coated surface of Faville-6 test sample prepared with Oakite HD-126 cleaning agent. Magnification 650X.

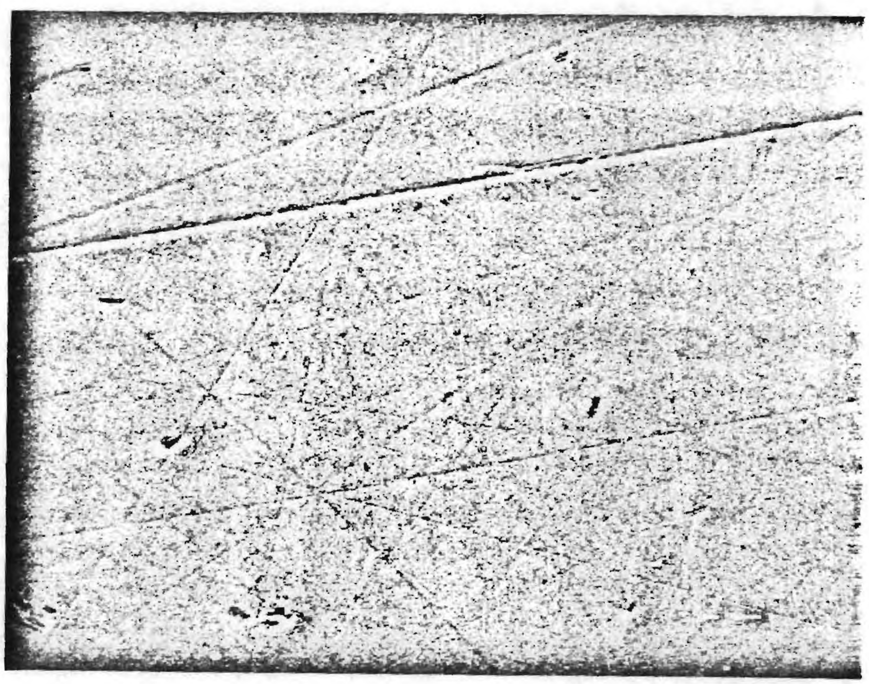

Figure 2.b. Same as above at 1200X. Note carbide pull out at isolated locations in the original surface of the test sample.

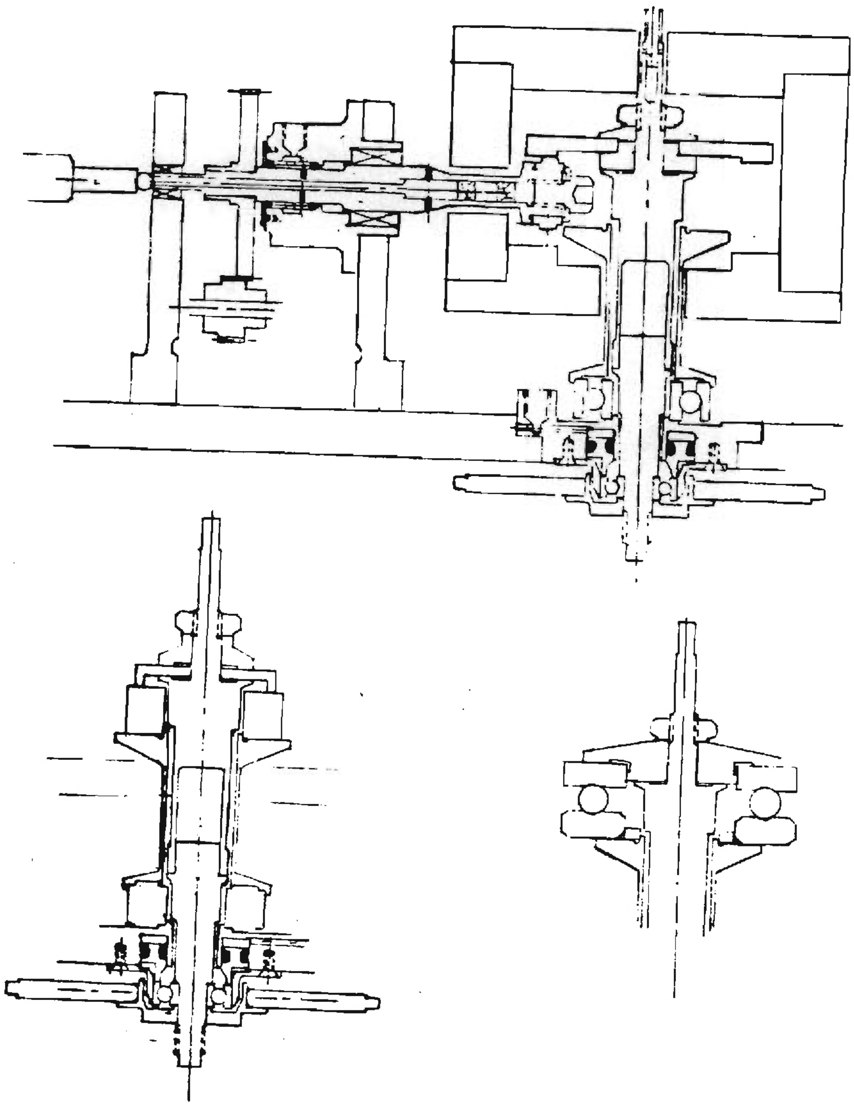

Figure 3. High temperature traction instrument.

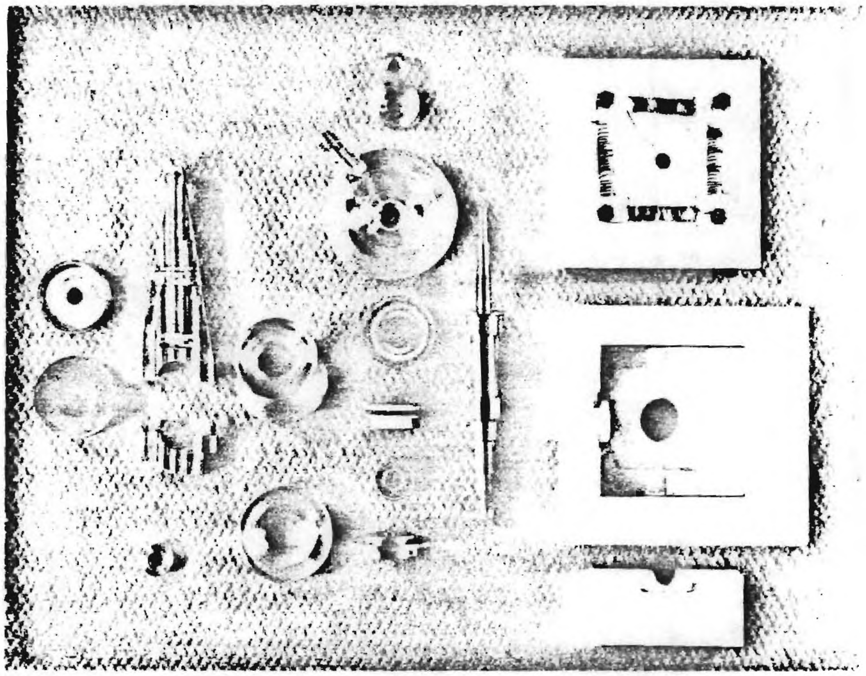

Figure 4. Traction instrument components.

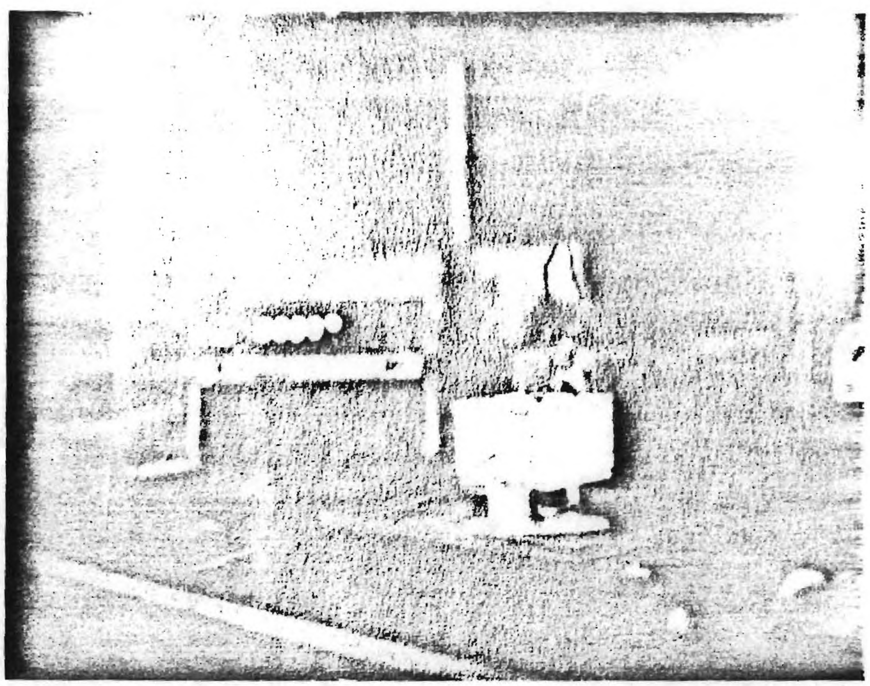

Figure 5. Traction instrument hot test.

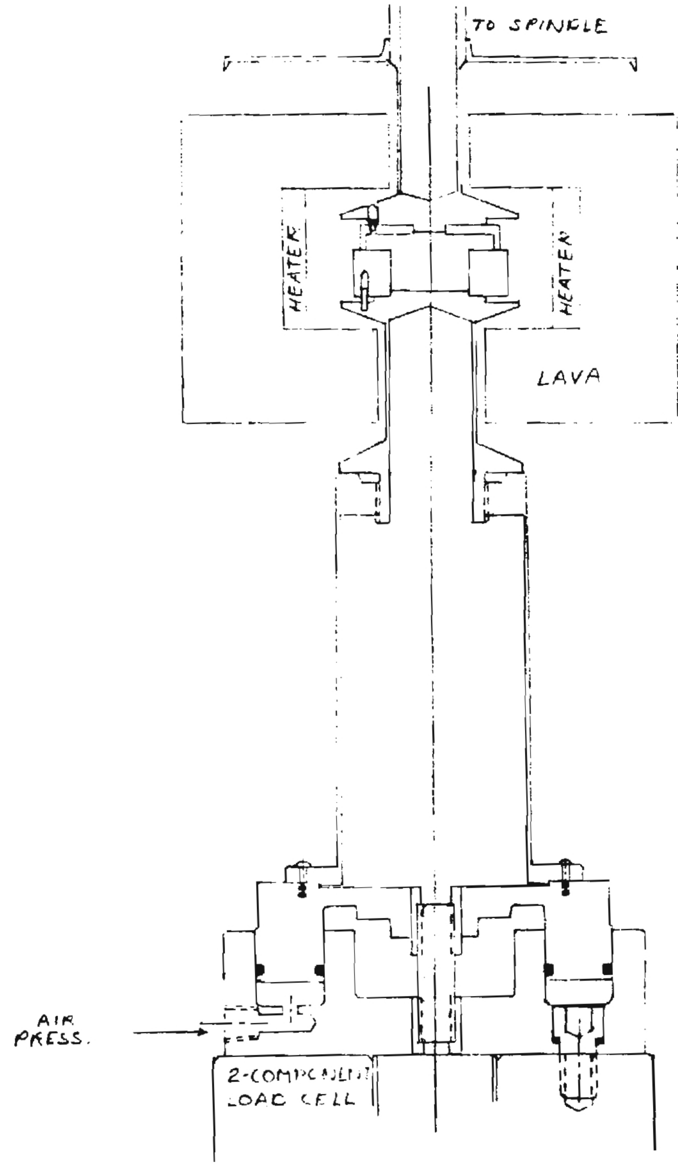

Figure 6. High temperature. Friction and life tribotester.

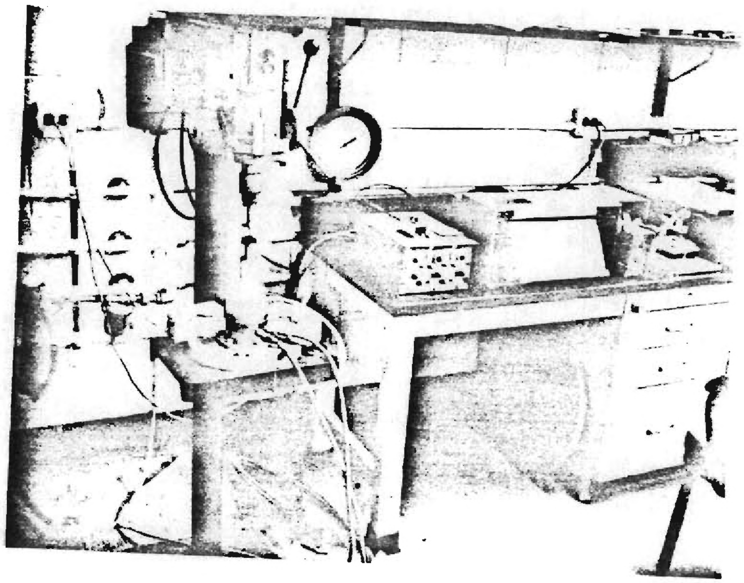

Figure 7. Friction and life tester.

FINAL REPORT

HUGHES AIRCRAFT P.O. NO. 04-406163-FS5

REACTIVE SPUTTERING OF TiN AND SOLID LUBRICANTS AND SHEAR STRENGTH TESTING OF SOLID LUBRICANTS

Co-Principal Investigators

S. Ramalingam, Professor

W. O. Winer, Professor

Prepared for

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December 1980

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December 1980

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Introduction

A research program on the reactive sputtering of titanium nitride, sputtering of solid lubricant materials and shear strength testing (traction in rolling contact) of solid lubricants was undertaken at the Georgia Institute of Technology to support Hughes Aircraft Company's efforts directed at the development of Solid Lubricated Rolling Element Bearings. S. Ramalingam and W. O. Winer of the School of Mechanical Engineering were the principal investigators at Georgia Tech and M. N. Gardos was the program manager at Hughes.

The Statement of Work No. FFD-442 dated 9 January 1980 issued by Hughes outlines in detail the work to be carried out at Georgia Tech on a "best efforts" basis.

This report covers the work conducted between 9 January 1980 and 31 December 1980 and is the Final Report on this contract. Portions of the work carried out and the results obtained were presented at the Critical Design Review (CDR) meeting held at Los Angeles in October 1980 as required in SOW No. FFD-442 dated 9 January 1980.

The work done and the results obtained which fulfill the requirements outlined as Tasks I to V in the SOW are presented here in six sections. A final section (Section 7) briefly summarizes the contents of the report.

1.1 Reactive DC-Magnetron Sputtering of Traction Specimens

A few sets of traction discs and bearing inner races together with about 50 balls (all 52100 steel) suitable for traction measurements were furnished by Hughes for titanium nitride coating at the start of the program. These samples were to be reactive sputter coated with titanium nitride (best effort at this point in time) and solid lubricant coated at Georgia Tech and Hughes respectively for subsequent use by MTI for traction measurements.

A special rotary jig that permits peripheral coating of traction discs and bearing inner races at a cathode-to-specimen distance of 7 cm was constructed. The rig was not equipped with a mask to ensure normal incidence of coating species during the coating of traction discs and bearing inner races. A stationary, perforated metal basket was constructed for the coating of AISI 52100 balls with titanium nitride. Uniform, total surface coating was not required for the balls.

1.2 Cleaning Procedure

The test samples to be coated were ultrasonically cleaned in xylene and acetone prior to vapor degreasing in trichloroethylene. Vapor degreased samples were washed again in acetone and dried in a blast of dry nitrogen. The test samples were then loaded into the vacuum system with minimal delay.

1.3 Coating Procedure

The traction discs and inner bearing races were coated individually in several runs. Balls were also coated in several runs with approximately eight to ten balls per run.

In a typical coating run, the sample to be coated was appropriately mounted in the coating chamber and the system was pumped to a pressure of 0.66 mPa. Standard argon back-filling and repumping schedules were used to minimize contaminants. Samples were then sputter-cleaned at a sputtering pressure of 2.66 Pa and voltage of 3 kV. The argon flow rate into the 12 inch diameter x 10 inch high sputtering chamber during sputter cleaning was five standard mℓ per minute. Test samples were typically sputter cleaned for 15 minutes.

Following sample cleaning, with the target shutter still in place, the magnetron sputtering head was turned on and the target was cleaned at a cathode current of one amp. System pressure at this stage is 122 mPa. Nitrogen was then admitted into the system while maintaining titanium sputtering and argon flow rate reduced to synthesize and deposit TiN. Shutter is then removed to initiate coating.

In a typical reactive sputter coating run, the stationary balls were coated for about ten minutes to obtain a coating thickness of 0.5 to 1.0 μm at a cathode current of 0.75 amp (5 cm target). The traction discs and inner races were coated for 25 to 30 minutes at a rotary speed of 30 rpm. Coating conditions employed were identical to these used for the balls.

1.4 Coating Characterization

Golden, yellow, visually perfect titanium nitride coats were obtained in all test samples. Taper-sectioned samples (coating on WC-C_O substrate) yielded a micro-hardness of 1900 Knoop (the value is lower than the theoretical 2700 Knoop due to substrate relaxation during loading in the micro-hardness test - verified by testing TiN-coated M-2 steel which yielded an even lower value due to greater substrate relaxation). X-ray diffraction patterns of titanium nitride coated $WC-C_o$ samples were obtained and they exhibited diffuse TiN peaks at 36.5°, 42.6° and 61.2° (2θ for copper K_α radiation, $\lambda = 1.542$ Å) corresponding to 111, 200 and 220 planes of titanium nitride. Some peak shifts, attributable to coating stresses, were also observed. The diffused peaks are most likely due to the very fine grain size in sputtered coatings.

1.5 Comments

The TiN-sputtered traction discs, inner races and traction balls are illustrated in Figure 17, page 34 of Hughes Semi-annual Status Report No. 3, covering the report period 1 August 1979 through 31 January 1980.

As of that time (January 1980) the cleaning and coating procedures were not developed enough to obtain the best possible adhesion. The coated, visually perfect test samples, probably did not possess high enough substrate-to-coating adhesion to withstand tractions with significant tangential components.

2.1 Reactive DC-Magnetron Sputtering of Friction and Wear Specimens

Preliminary investigations on titanium nitride coated balls, 32mm in diameter, carried out at Georgia Tech demonstrated the ability of very thin hard coats to withstand Hertzian contact pressures of 1.343 GPa for 10^6 cycles in a lubricated rolling contact test. An unformulated, low viscosity mineral oil with a viscosity of 24 cst at 37C and 3.7 cst at 98C was used in the test which ran at 1 m/sec and room temperature ($\simeq$ 24C). No surface asperity interaction occurred. Film failure was however observed to occur at sliding contacts following some 30,000 cycles. If the non-distortive coating process is to be viable to produce hard-coated rolling bearing elements improvements in coating-to-substrate adhesion are essential. Task II is directly concerned with process development and identification of coating parameters to obtain best possible coating adhesion in rolling contact bearing elements.

Successfully withstanding the sliding and rolling contact wear tests defined in Task II of Hughes purchase order no. 091837 dated 9 January 1980 is to be the basis for judging the adequacy of coating-to-substrate adhesion.

2.2 Process Development

The major problem in titanium nitride-to-substrate adhesion is thought to arise from the lattice mismatch involved and the coefficient of expansion mismatch between TiN and steel. Studies carried out at Litton (Appendix A, Hughes Semi-annual Status Report No. 2, February

1979 to July 1979) suggest that the surface damage accompanying commercial ball production practice (ball or race grinding and lapping) may also lead to coating adhesion problems. Coating process development work was undertaken and carried out at Georgia Tech, to overcome these difficulties. The following sections summarize the work carried out and results obtained.

Very poor adhesion is easily detected since the coating spalls off the surface. Coatings with a little better adhesion exhibit a network of cracks usually visible to the naked eye. Better bonded coats (not necessarily adequate) do not exhibit the crack network and withstand the common peel test (Peeling force transmitted with the aid of a scotch tape) without failure. Sliding wear tests are then the first arbiters of coating adhesion. Task II requires disc-to-disc, flat, LFW-6 tests (test geometries illustrated in Figures 4 and 5, pages 22, 23, of Hughes Semi-annual Status Report No. 2, 1 February 1979 to 31 July 1979) at 70 rpm with a contact stress of 3.5 MPa (500 psi) for 30 minutes with n-hexadecane as the lubricant to document adequate coating adhesion. Coatings surviving the sliding contact tests are candidates for rolling contact thrust washer tests (LFW-6 tests, illustrated in Figures 4 and 6, pages 22-23, Hughes Semi-annual Status Report No. 2, 1 February 1979 to 31 July 1979). Coatings surviving thrust washer rolling contact tests at 500 rpm and 450N (100 lb) thrust load for 30 minutes with n-hexadecane lubricant are considered to be satisfactory for bearing service. (These room temperature tests subject the rolling elements to a hertz contact stress of 1.5 GPa).

Magnetron-sputtered titanium carbide coatings were found to crack and spall off in an earlier part of the present program (See Figures 90 to 96, pages A-69 and A-70 of Hughes Semi-annual Status Report No. 3, 1 August 1979 to 31 January 1980). Similar problems were expected in magnetron reactive sputtering of titanium nitride on hardened steel surfaces. Process development activity was aimed at overcoming this specific problem.

Magnetron-sputtered titanium carbide coatings were successful on beryllium substrates for the construction of gas bearings (R. P. Riegart, Technical Report No. AFML-TR-78-192, WPAFB, Ohio 45433). The good adhesion of TiC on beryllium has been attributed to the high oxygen content of the substrate material. Work carried out at NASA-Lewis is in agreement with this view for other hard coats such as Mo_2C.

2.3 Pre-Oxidation to Enhance Coating Adhesion

An initial series of coating process development studies were carried out where disc-on-disc flat test samples were initially oxidized and then reactively sputtered. Hardened 4340 Steel and 52100 Steel test samples were used. Test sample geometries are specified in the Barden Corporation drawings numbered Z1127 and Z1138.

For coating trials, the test samples were initially diamond polished with 7 μm diamond paste in a Struer's automatic polisher with a light load (of the order of 5N (0.5 Kg). Following polishing to a mirror finish, the samples were washed, degreased, washed and dried as described in Section 1.2 of this report. The test samples were pre-oxidized in an oven for various times at three temperatures

(10 min., 30 min. and 60 min., at 150C (300°F), 260C (500°F) and 426C (800°F). The procedure used is comparable to that followed in NASA-Lewis to enhance coating adhesion.

Pre-oxidized samples were reactively sputtered with TiN using the procedures described earlier (Section 1.3). Trials were also conducted in which the sputter cleaning step was not used or was used for shorter times. Every one of the trials was a failure. Spalling and/or network cracking was the mode of film failure.

Pre-oxidation treatments were found to be of no value in enhancing the adhesion of reactively sputtered titanium nitride to hardened 4340 and 52100 Steel substrates. Since coated samples were poorly bonded, friction tests were not carried out.

2.4 Elevated Temperature Sputtering

An announcement from Battelle-Geneva (received from T. Dow of Battelle-Columbus) reported on the successful reactive sputtering of titanium nitride at substrate temperatures of 500°C. The work is attributed to Drs. Zega and Talmor of Battelle-Geneva. This and the need for substrate heating well-documented in works on ARE (Activated, Reactive Evaporation) were the impetus for examining the suitability of sputtering on hot substrates to improve film adhesion.

To examine the viability of hot-coating, a heating stage was constructed (with thermocouple for temperature measurement) with a pancake resistance heater. Test samples (Drawing Z1138) prepared as described in Section 2.3 were placed on the hot stage and the vacuum system was pumped down to 7 mPa (5×10^{-5} mm of Hg) before heater power was turned on. This served to prevent oxidation of the surface to be coated. The test samples were sputter cleaned and sputter coated while hot. The sputtering procedure was identical to that described in Section 1.3.

Several coating runs were carried out between 200C and 540C (400°F and 1000°F) in 100°F (55C) increments. The upper temperature limit is imposed by the loss of temper of hardened 52100 (and 4340) steel.

Some improvement was noticeable in all hot-coated test samples. In every instance, coated samples exhibited a crack network (visually recognizable). Film cracking could not be prevented even with very low coating rates. Large scale film spall was not observed. Since film cracking was considered to constitute failure, friction and wear tests were not carried out on hot-coated test samples.

2.5 Surface-treatment (Chemical) before Sputtering

Films laid on well-cleaned substrates should at least yield a van der Wall binding. Bond strength calculations at a mean film-to-substrate distance of 5 to 10 Å (using van der Wall cube law) yield bond strengths of the order of 200 MPa to 100 MPa (30,000 to 15,000 psi) sufficient to permit contact stresses of 1.4 GPa (200,000 psi) in the presence of friction coefficients of the order of 0.05 to 0.10. These numbers would imply that reactive sputtering ought to be able to yield adequate film bond strength, if the surfaces to be coated are sufficiently clean (i.e., free from oxides, physically or chemically adsorbed atomic species, etc.).

This reasoning would attribute the failure of pre-oxidation to failure at the metal-oxide or oxide-coating interface or to failure within the oxide itself (SEM does not have adequate resolution to test these postulates). Failure of the elevated temperature coating would be attributed to mismatch in dimensions during cooling.

Coating adhesion problems of the type encountered here are also common in metal plating (by electrolytic means). A variety of surface cleaning and treatment techniques involving proprietary chemicals is common in the plating industry. Coating trials were hence undertaken to examine, if suitable chemical surface treatments can be found to overcome the adhesion problem.

A variety of alkaline chemicals and etching agents as well as proprietary chemicals were tested. A 'Metal Conditioner' marketed by William T. Bean and Co., Chicago, Illinois, has been found to yield reproducibly good reactively sputtered film adherence.

Following degreasing in acetone and drying in a blast of nitrogen the surface to be coated is immersed for a few seconds in the 'metal conditioner' washed again and then dried in a nitrogen blast. Samples loaded in vacuum are then brought to a pressure of 0.66 mPa, and allowed to degas for a few minutes. Following sputter cleaning, the TiN film is laid as described in Section 1.3.

Well-bonded, titanium nitride films are produced routinely. Figure 1 illustrates a SEM micrograph of a selected region of the coated test sample (disc-to-disc, flat). The coating is free from nodular growth and is of uniform thickness. The upper surface of the coating reproduces the base surface finish including five scale scratches produced by 7 μm diamond polishing.

A circular region without TiN coating and a pit at the center of the TiN-free region are also seen. The pits and the reaction product contaminated region are produced in the course of the surface treatment with the proprietary 'metal conditioner'. The metal conditioner (supplied by Wm. T. Bean Co., Chicago, Illinois) is a phosphonic acid-base solution, which, in the course of neutralization (with water), following surface treatment produces pits whenever the neutralizing fluid contains chlorine ions. An 'as surface treated' test sample surface is shown in Figure 2.

The 'metal conditioner' does not produce etch-relief. Pits are produced but the pit spacing does not correlate with the mean carbide spacing in hardened AISI 52100 Steel. The observed pits are therefore attributable to improper 'metal conditioner' neutralization following chemical surface treatment.

Pitting is a common problem in surface preparation for electrolytic plating when commercial water is used. The usual remedies are to use 'anti-pitting' chemical additives or to use distilled water of high resistivity (demineralized, ion-free water). The pitting problem is controllable and can be eliminated without too much difficulty.

Since the primary goal of the present program is to produce well-adherant coatings and to evaluate the tribological properties, a decision was made to proceed with the evaluation program, even with pitted films. Two justifications are offered for this course of action: (a) pitting is curable, and (b) tribological evaluation of films with pit defects constitute a more severe film adherence test.

2.6 Disc-on-disc, Sliding Wear Tests and Titanium-nitride Coating Performance

The sliding contact friction tests (LFW-6 type disc-to-disc; flat with Barden Z1137 top plate; Barden Z1138 bottom plate) required 300 kPa (50 psi) nominal contact stress at 70 rpm with n-hexadecane as the lubricant. A more severe contact stress (600 kPa = 90 psi) was used in the test program to subject the coating-to-substrate interface to a higher level of distress. The normal load and frictional torque were continuously measured during the test (30 minute duration) with a quartz crystal dynamometer.

The friction coefficient that prevailed during the test was determined by assuming that the contact stress is uniformly distributed over the nominal contact area. Hardened steel-to-steel sliding contact tests, under identical test conditions, were also carried out to establish a reference base.

The measured coefficient of friction as a function of time for both series of tests are shown in Figure 3. Noticeable wear track with considerable metal loss was observed in the steel-to-steel test. A burnished track without large scale material loss was observed in the TiN-to-TiN sliding contact. A steady state friction coefficient of 0.12 was measured in the TiN-on-TiN tests.

SEM micrographs obtained from two selected regions in the wear track of TiN-coated 52100 Steel base plate (Z 1138) are shown in Figure 4. Film surface structure is virtually unchanged (compare with Figure 1). Minor separation of titanium nitride at the boundaries of pitting artifacts is observable. The loose debris produced at these locations is trapped between sliders and plows a groove on the contact surfaces. Despite the higher local stresses, there is little material loss in the vicinity of the plowed tracks. Contiguous film loss is observable only between closely spaced pit artifacts.

The titanium nitride reactive sputtering practice developed is therefore considered to be satisfactory for low contact stress, sliding contact service. Pitting artifact elimination is desirable (work is in progress to eliminate pitting during surface preparation).

Rolling contact tests were undertaken and carried out following the sliding contact tests. They are described in a subsequent section.

3.1 Magnetron-sputtering of Low Shear Strength Solid Lubricant Layers

The present program is aimed at the development of solid lubricated rolling element bearings. Soft coat overlays on hard-coated bearing elements are desired. Task IV requires the magnetron sputtering of WSe_2/In/Ga alloy (Westinghouse Compact) using targets furnished by Hughes Aircraft Company. The mini-magnetron constructed and demonstrated by Georgia Tech, was to be used.

Tungsten Selenide/In/Ga discs (Westinghouse compact) 50 mm in diameter x 3 mm were furnished by Hughes (courtesy of R. McConnell, AFML, WPAFB, Ohio) for the fabrication of mini-magnetron targets. The discs were pressed and sintered bodies.

Georgia Institute of Technology's mini-magnetron target is an annular disc 50 mm O.D. x 20 mm I.D. x 3 mm. The discs furnished had to be machined to produce the inner hole. A collect fixture was designed and built to hold the discs on the 50 mm O.D., to drill the hole required. The sintered targets did not possess adequate strength and hole drilling resulted in target disc disintegration. Mini-magnetron system, hence, could not be used in the present program.

The planar magnetron used for reactive sputtering was therefore modified and adopted to permit magnetron sputtering of WSe_2/In/Ga alloy. Satisfactory magnetron sputtering parameters had to be and were established empirically.

3.2 Westinghouse Compact Sputtering Parameters

Planar magnetron sputtering of WSe_2/In/Ga alloy can be carried out under the following sputtering conditions:

Sputtering voltage	745 VDC
Sputtering current*	100 ma
Sputtering pressure	2.0 Pa (15 μm of Hg)

*Cathode current is limited by possible target melting. Disc targets cracked in use. Disc targets were bonded to a copper backing plate with silver epoxy (conductive epoxy cement).

In a typical solid lubricant coating run, the argon flow rate was maintained at 3 std. mℓ per minute. Following each run, odors of selenium vapor could be smelled strongly in the sputtering chamber as well as in the room. Apparently the high vapor pressure of selenium is responsible for this (This observation served to limit the power input to the WSe_2/In/Ga target).

3.3 Sputtered Film Structure

Figure 5 illustrates the edge of a spalled film (produced in early sputtering trials) of solid lubricant. Feature-free and superficially homogeneous films are produced. As in the case of titanium nitride coatings, the films produced are nodule-free. The upper surface of the coating reproduces the base surface finish of the coated body.

The Westinghouse compact is a polyphase material. Figure 6A is a low magnification view of the fracture surface of a fractured target (remnant from the attempt at drilling). The target is not homogeneous and as shown in Figures 6B and 7, at least three distinct phases in fibril, spheroid and flake morphology could be detected.

to 10 μg) and overlaid with an additional 200 nm of WSe_2/In/Ga film.

Sliding friction tests were also carried out on bare steel top plates coated with Westinghouse material running against TiN-coated base plates. A last series of tests used TiN-coated top plates against TiN and WSe_2/In/Ga coated base plates. In all cases, each coating, when used had a thickness of 200 nm.

The test results obtained are presented in Figures 8 and 9. Measured friction coefficient is presented as a function of test time. In all cases a slight 'running-in' period can be detected. The duplex coatings (soft coat overlay on hard substrate) yielded lower friction coefficient. Westinghouse compact material in the sputtered thin film is found to be a solid lubricant coating ($\mu \simeq 0.07$ to 0.075).

Test data shown in Figure 9 can be interpreted to indicate the possible existence of an endurance limit in the case of a solid lubricant film. It is not yet clear if the increase in friction coefficient with time is due to <u>wear</u> of the soft coat or due to less than perfect adhesion of the soft coat to the substrate (There is as yet no adequate adherence test for soft coats. Peel test with scotch tape is inadequate due to the inherently low strength of the soft coats). The less pronounced time-dependent change in friction coefficient in the case of duplex-coated (soft coat overlay on TiN, Figure 8) can be attributed to the transfer of the soft layer back and forth between counterfaces. The pit artifacts in TiN can serve in this process as a 'reservoir' for the soft film material.

3.5 SEM Observations

A selected SEM micrograph from a duplex-coated test sample is shown in Figure 10A. This represents the virgin surface of the coated sample. The pitting artifact, TiN film and the WSe_2/In/Ga alloy film are seen. A comparable region from the sliding friction test track is shown in Figure 10B. The pit artifact serving as a local solid film reservoir is shown in Figure 10C. Isolated locations where the soft coat has been removed is seen in Figure 10D.

As in the case of TiN-coated test samples, there has been small scale separation of the hard coat at the pitting artifacts. The hard, loose debris is responsible for the wear track running diagonally across the micrograph. The debonding of the soft coat parallel to the wear track (wear groove) suggests that the loose debris (TiN) formation is the most probable cause for the loss of soft film at this location.

A comparison of the virgin surface of the soft coat (Figure 10A) with the soft coat surface in the friction track (Figure 10D) suggests that transfer phenomena could well have occurred during sliding contact.

The soft coat sliding friction test results and SEM observations presented testify to the general adequacy of the sputtering practice developed. Though some benefits are apparently attainable by retaining the pitting artifact in the TiN film, it is most probably not desirable since it tends to cause soft coat film loss. The need to eliminate the pitting artifact in TiN film is considered to be real and is being followed up.

4.1 Rolling Contact Studies - Hard Coats

Task II requires rolling contact thrust washer tests on coated bearing races at a hertzian contact stress of one GPa with n-hexadecane lubrication. The satisfactory sliding contact tests (LFW-6, Disc-to-Disc) were therefore followed up with LFW-6 tests on coated thrust bearing races. Tests lasting 30 minutes were carried out at 500 rpm using a full complement of balls. A Delrin cage was fabricated and used.

One race of the thrust bearing (as ground bearing race with no polishing) was cleaned and reactively sputtered with titanium nitride. The procedures and coating practice employed were identical with those used for the LFW-6 sliding contact tests.

The titanium nitride coated lower thrust bearing race was reassembled with a full complement of balls and the Delrin cage mentioned earlier. The assembly was tested in a LFW-6 test simulation rig with a thrust load of 450N which corresponds to a hertz contact stress of 1 GPa. The reactively sputtered TiN coats withstood the 30 minute test duration without coating failure. Figures 11 and 12 document the coating structure before and after the rolling contact test.

Figure 11A shows the topography of the 'as reactively sputtered' ball race. Coarse grinding marks and a pitting artifact produced during surface preparation for sputtering are seen. Figure 11B shows topography of the ball race at a location where the contact had primarily been one of nearly pure rolling. There is little evidence of coating distress. A ball race location with some sliding is shown in Figure 12A. Unidentified debris filling of pitting artifact is seen. Evidence indicative of coating surface distress due to sliding and some transfer can be seen in Figure 12B. Even at locations of pitting artifact, there has been no film failure.

The uncoated balls acquired a golden yellow hue during the test indicative of TiN transfer. Ball surface structure following the test run are shown in Figure 13. Micrographs reveal that some transfer has occurred. A part of the transferred material must most certainly be from the Delrin cage. The SEM examination did not use x-ray micro-analysis to evaluate the relative amount of polymer and TiN in the transferred material.

4.2 Rolling Contact Tests in the Traction Test Rig

The concentrated contact simulator design and built to measure dry rolling traction characteristics of solid lubricant films (Figure 14) was also used to test the adhesion of reactively sputtered films in pure rolling contact tests. Hardened 52100 traction discs and rollers were TiN coated as described in earlier sections. Rolling contact tests (at 2 m/sec., 1.07 GPa hertz contact stress) carried out in the concentrated contact simulator did not cause film failure. The contact surface in this instance was diamond polished (7 μm diamond). Since coatings in the ground ball race were able to withstand comparable hertzian contact stresses (LFW-6 tests on thrust bearings), the results obtained were quite as expected.

5.1 Shear Traction Measurements of Solid Lubricant Films

Task V of the current program requires shear strength measurements on candidate solid lubricant materials. Hughes-applied MoS_2 and Georgia Tech-applied solid lubricant coatings were to be evaluated. Shear traction measurements at pressures to 1 GPa (160 ksi) are required to model solid lubricant film behavior in rolling contacts. Traction measurements in pure rolling contact and in rolling with a known slide-roll ratio are required.

5.2 Concentrated Contact Simulator

To facilitate the measurement of dry rolling traction characteristics of solid lubricant films, a new concentrated contact simulator was designed and built. It is illustrated schematically in Figure 14. The configuration employed is that of a spherical crowned roller running against a flat traction disc. The simulator allows recording of the traction force, while the slide-roll ratio is continuously varied. The slide-roll ratio, $\Sigma = \frac{2(u_1 - u_2)}{(u_1 + u_2)}$ is the ratio of sliding to rolling velocity where u_1 and u_2 are the two surface velocities. The rolling speed (average speed of both elements) is maintained essentially constant. The normal load is applied by dead weight to produce a circular, elastically deformed contact about 0.1 mm in diameter. The pressure distribution is hertzian with maxima of 0.79 GPa and 1.07 GPa.

5.3 Shear/Traction Test Data

Traction discs and rollers were sputtered with MoS_2 and

Westinghouse compact material at Hughes and Georgia Tech respectively using RF sputtering and magnetron sputtering. Two different solid lubricant film thicknesses (400 nm and 1500 nm) of MoS_2 were tested. Two test samples coated with Westinghouse material, both with a solid lubricant film thickness of 400 nm, were tested. The test conditions and the test results obtained are shown in Figures 15 to 19.

In all cases, the sputtered coatings (MoS_2 and WSe_2/In/Ga) debonded from the roller immediately on the initiation of the test run. In all the tests, where the sliding velocity is less than three percent of the rolling, the disc coating remained in situ without damage. The traction test data obtained yield traction coefficients of approximately 0.3 and 0.20 for MoS_2 and WSe_2, respectively when the sliding velocity is of the order of, or greater than four percent of, the rolling velocity.

Compared to oil lubricated elements, the operation of the solid lubricant coated elements was quite noisy. Approximate number of revolutions of the roller at the time of measurement is indicated in each figure. The level of traction is found to increase in the first 2000 revolutions of operation to level off to become nearly constant.

5.4 Shear/Traction Test Data - Burnished Films

Although Task V did not call for shear/traction test data on burnished films of solid lubricants, traction measurements were also made with burnished films of MoS_2 and Westinghouse compact material. The solid lubricant was rubbed on to the test surfaces with a paper tissue. The test results obtained are shown in Figures 20 to 22.

Operation was quiet and tractions reduced in comparison with the sputtered films.

Figure 22 presents results from a pair of TiN sputtered elements which received a burnishing of WSe_2/In/Ga alloy. Traction was comparable to uncoated steel with burnished films of Westinghouse compact material. After 3000 revolutions of the roller, the sputtered titanium nitride was partially removed from the roller and from areas of the disc which experienced the highest amount of sliding.

5.5 SEM Observations - Sputtered and Burnished Solid Lubricant Coatings

The appearance of the solid lubricant coated traction disc before and after use in the concentrated contact simulator is as shown in Figure 23. The film is free from nodular features and its upper surface faithfully reproduces the topography of the original disc surface. In pure rolling contact film damage is minimal as is apparent from Figure 23B. A low magnification view of the traction disc coated with the Westinghouse material is shown in Figure 24A. The appearance of the MoS_2 coated disc is similar. In both cases, debonding occurred at inner and outer radii of the traction disc where the sliding velocity exceeded (approximately) three to four percent of the rolling velocity. Figures 24B and 25A illustrate the appearance of the film-debonded regions at the inner and outer radii respectively. SEM observations show that the appearance of the MoS_2-coated traction disc is quite similar to that of Westinghouse material coated traction discs at corresponding locations.

Sputtered films of solid lubricant materials require further development in substrate-to-coating adhesion, if they are to withstand significant slide-roll ratios during nominally rolling contact.

6.1 Other Support Activities

To support the non-distortive hard coating technology development and to assist the development of solid lubricant coating techniques, some additional work involving ball coating fixtures, sputtering process development, analysis of coating flux in sputtering system, etc., were carried out. The following sections briefly summarize these activities.

6.2 Fixture Development for Ball Coating

Earlier work involving RF sputtering of MoS_2 at Hughes indicated a systematic variation in tribological properties of sputtered films with substrate orientation during coating. Sputtered flux arriving normally on to the test sample surface was found to yield the lowest friction coefficient. The X-ray diffraction studies carried out on reactively sputtered films of TiN (at Georgia Tech) have also been found to produce a preferred crystallographic texture in the coated film. Special ball coating fixtures which only permit normal incidence of coating flux was therefore deemed necessary to take advantage of the preferred texture produced during solid lubricant sputter coating.

In consultation with M. Gardos and R. L. Christy of Hughes, a motorized ball coating fixture has been design and built. Preliminary sputtering trials have been carried out. Detailed analysis of the coated test samples has not been carried out.

6.3 Duplicate Coating Chamber Development

Analysis of the molybdenum disulfide coating produced at Hughes using sophisticated structural analysis techniques (Rutherford Back-scattering studies carried out at NRL, Washington, by J. Hirvonen and C. Carosella) indicated the possibility of sputtered film contamination by re-sputtering of earlier coatings (off the chamber walls and fixtures). To offset the possibility of similar cross-contamination in reactive sputtering of titanium nitride and in the sputtering of solid lubricant films, a duplicate sputtering system was felt to be necessary both by the Georgia Tech principal investigators and by the Program Monitor at Hughes (M. Gardos). In conformity with this perceived need, a new sputtering system has been built and the ball coating fixture has been installed in this system.

6.4 Coating Flux Studies

Planar and mini-magnetron sputtering systems are extended source coating devices. Coated film thickness obtained under a fixed set of operating conditions is position-dependent. Subsequent ball coating activities require a knowledge of coating flux distribution as a function of substrate location within the sputtering chamber. To determine this, a series of titanium sputtering studies were carried out. A precision surface profile recorder was used to measure the coated film thickness. Suitably masked witness coupons produced by placing the coupons at a fixed target-to-substrate axial distance and variable radial distance were used to determine the coating flux variation with radial distance.

A Hall probe was used to measure the magnetic field strength on the target surface. Assuming that only the horizontal component of the magnetic field is effective in generating the electron trap of the magnetron, coating flux distribution was calculated as a function of radial location (at a constant target-to-substrate axial distance). The extended source computations carried out are in good agreement with the experimentally determined radial distribution.

6.5 Effect on Ion Implantation in Improving Coating-to-Substrate Adhesion

In course of the Program Review Meeting at Dayton (1979), arguments were put forward to examine the utility of ion implantation (Ti ions) in enhancing the titanium nitride to hardened steel adhesion. The Ion-implanted LFW-6 test samples produced at NRL were reactively sputtered with Titanium nitride at Georgia Tech to assess the benefits of ion implantation. Trials carried out so far indicate that little particular benefits can be realized by ion implantation prior to reactive sputtering. Reactively sputtered, ion implanted test samples spalled off as readily as those not implanted.

6.6 Test Samples for Stoichiometry Analysis

X-ray diffraction studies of defect compounds and of sputtered poly-phase materials that may possess amorphous structure (WSe_2/In/Ga alloy) are prone to considerable error in measurement, analysis and interpretation. Rutherford backscattering techniques are better suited for this analytical task.

Carbon-coated silicon test samples and beryllium test samples were furnished to Georgia Tech (by Hughes and NRL respectively) for reactive sputtering with titanium nitride and magnetron sputtering with the Westinghouse material. Test samples were coated (best effort) and sent to the Naval Research Laboratory for analysis. Some stainless steel test coupons suitably coated with TiN and Westinghouse material have also been furnished. Detailed test results are awaited.

7.1 Summary

1. Rolling bearing elements can be non-distortively hard-coated by reactive sputtering of titanium targets in an argon-nitrogen ambient with a suitably designed Magnetron sputtering head. Golden yellow, titanium nitride hard coats with satisfactory stoichiometry and hardness can be reproducibly deposited on selected substrates at high cathode current densities (of the order of 40 ma/cm^2 or better) in a good vacuum (better than 0.13 Pa).

2. Cleaning and surface treatment procedures have been empirically identified that yield well-adherent thin coats on hardened steels (52100 steel) capable of withstanding high hertzian contact stresses (to 1 GPa). The surface treatment procedure developed produces pitting artifacts which do not affect the hard coat performance. The artifacts are curable and should be eliminated eventually.

3. Titanium nitride hard coats on hardened 52100 steel yield a sliding friction coefficient of 0.12 at a contact stress of 61.5 kPa when n-hexadecane is used as the lubricant. Despite the presence of pitting artifacts, the hard coats are durable in low contact stress sliding contact.

4. The hard coats are also able to withstand hertzian contact stresses to 1 GPa at 1 to 2 m/sec rolling velocity without noticeable film damage. Thrust bearing ball races in the 'as ground' condition can be satisfactorily hard coated by reactive sputtering and do not fail even with n-hexadecane as the lubricant (Low lambda ratio rolling contact tests).

5. Westinghouse compact material can be satisfactorily sputtered in a planar magnetron. Sputtering current density is limited to values of the order of 5 ma/cm^2 by the tendency of the pressed and sintered target to crack during operation.

6. Sputtered films of the Westinghouse compact material are solid lubricants with a sliding friction coefficient of the order of 0.07 to 0.075. Satisfactory film adherence is obtained at the sputtering pressure used (2 Pa), but further lowering of sputtering pressure would appear to be desirable.

7. Traction measurements on MoS_2 and Westinghouse compact sputtered traction discs show both these materials to be solid lubricants. Limiting traction coefficients of the order of 0.2 to 0.3 are obtained with solid lubricants when the sliding velocity is of the order of four percent of the rolling velocity.

Improvements in coating to substrate adhesion is desirable, if the solid lubricant coatings are to withstand significant amounts of sliding.

8. Despite the initial polyphase structure, Westinghouse compact apparently produces a homogeneous solid lubricant film when sputtered. Structural analysis of the sputtered film is desirable.

9. Duplex coatings (soft overlays on hard coats) are easily produced by sputtering. Duplex-coated bodies exhibit frictional properties in sliding contact characteristic of the soft overlay coats.

10. Film durability and film property (tribological) studies as a function of sputtering conditions and film thickness are desirable.

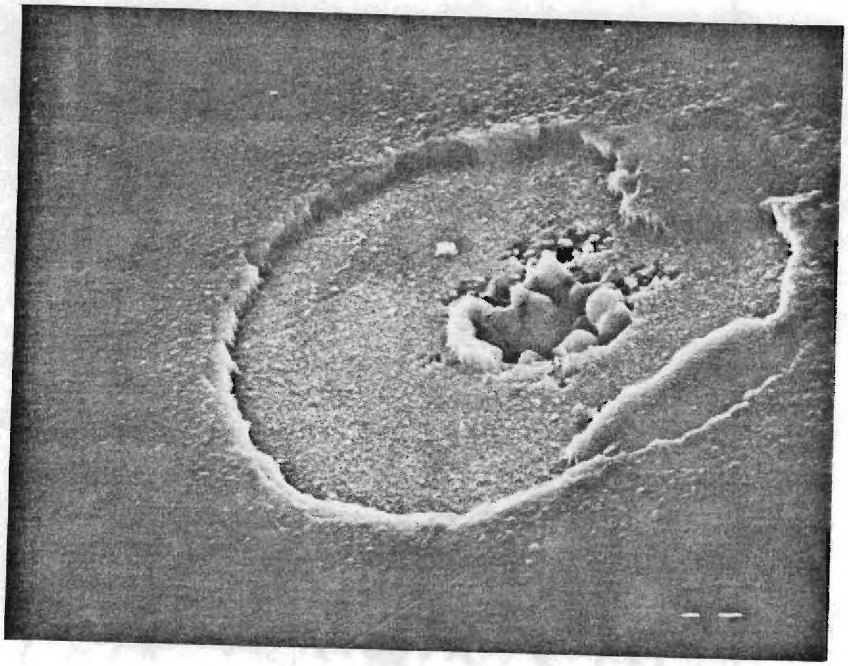


Figure 1. Representative TiN-coated surface With pit artifact. 2100 X.

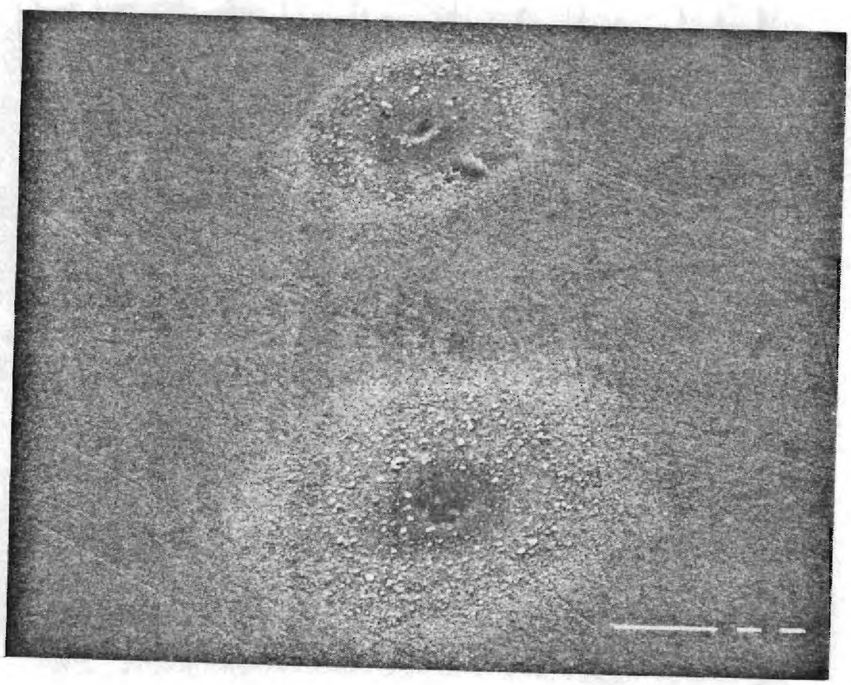


Figure 2. Pitting artifacts in metal conditioner treated surface of 52100 steel. 1340 X.

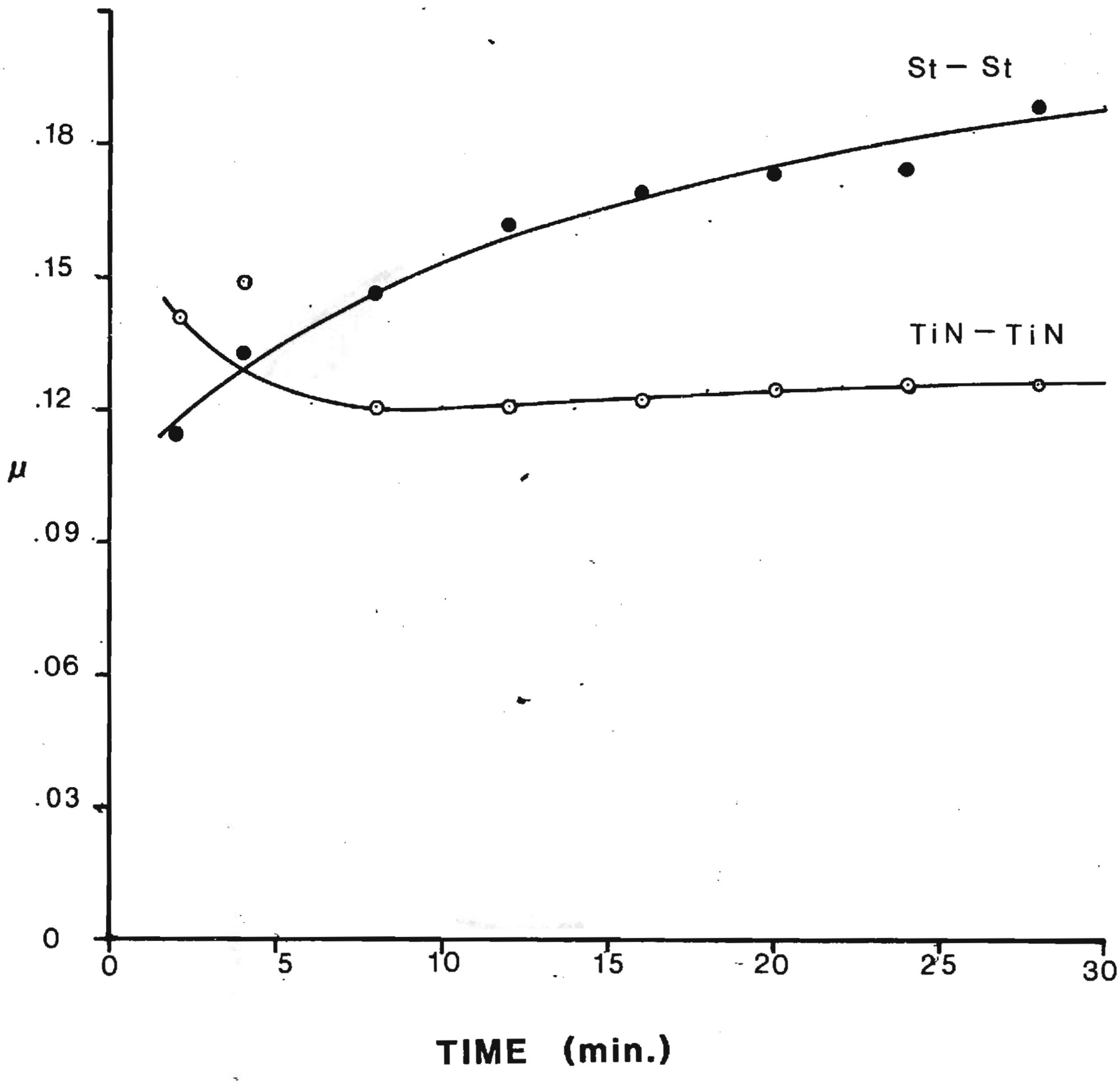
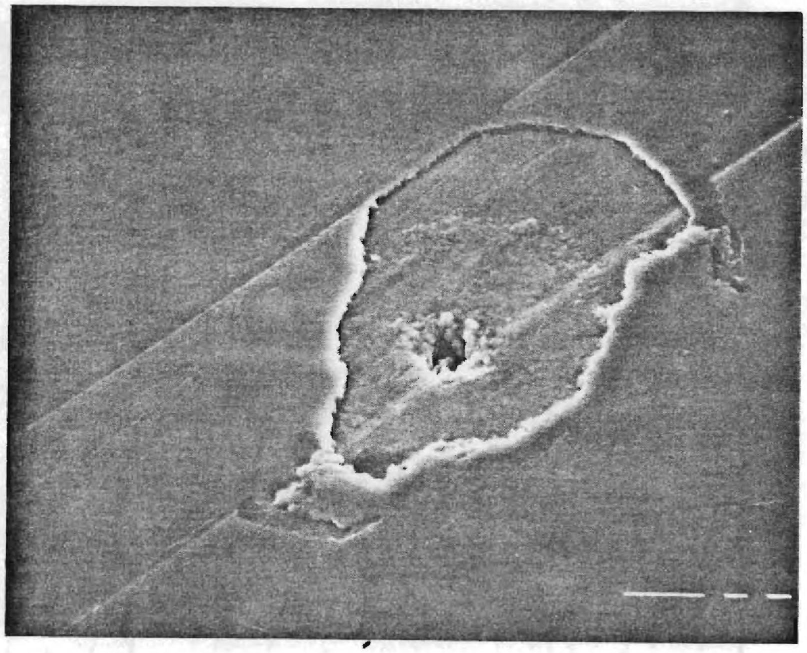
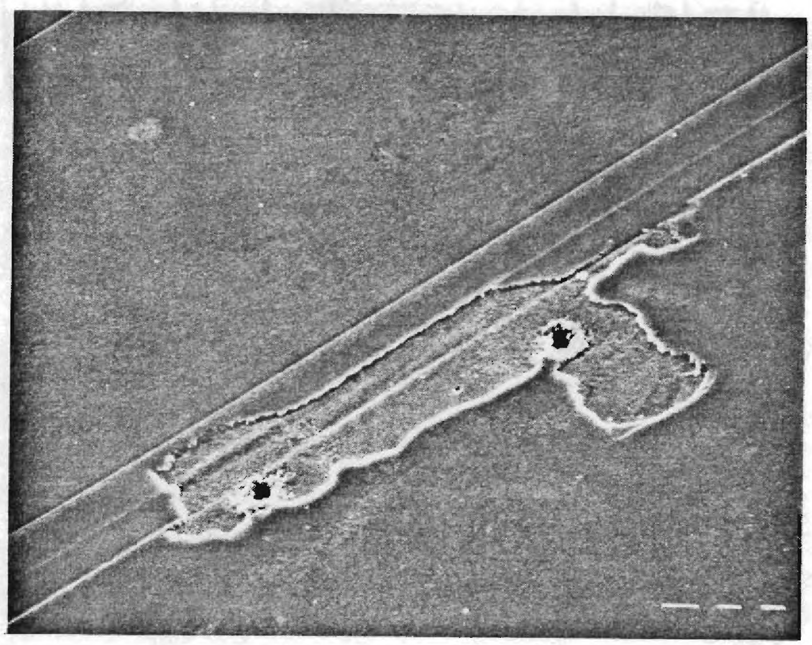


Figure 3. Measured coefficient of friction as a function of time in the LFW-6 disc-to-disc sliding friction test. For test conditions see Para 2 of Section 2.2. Test data for steel-on-steel and TiN-on-TiN are presented.

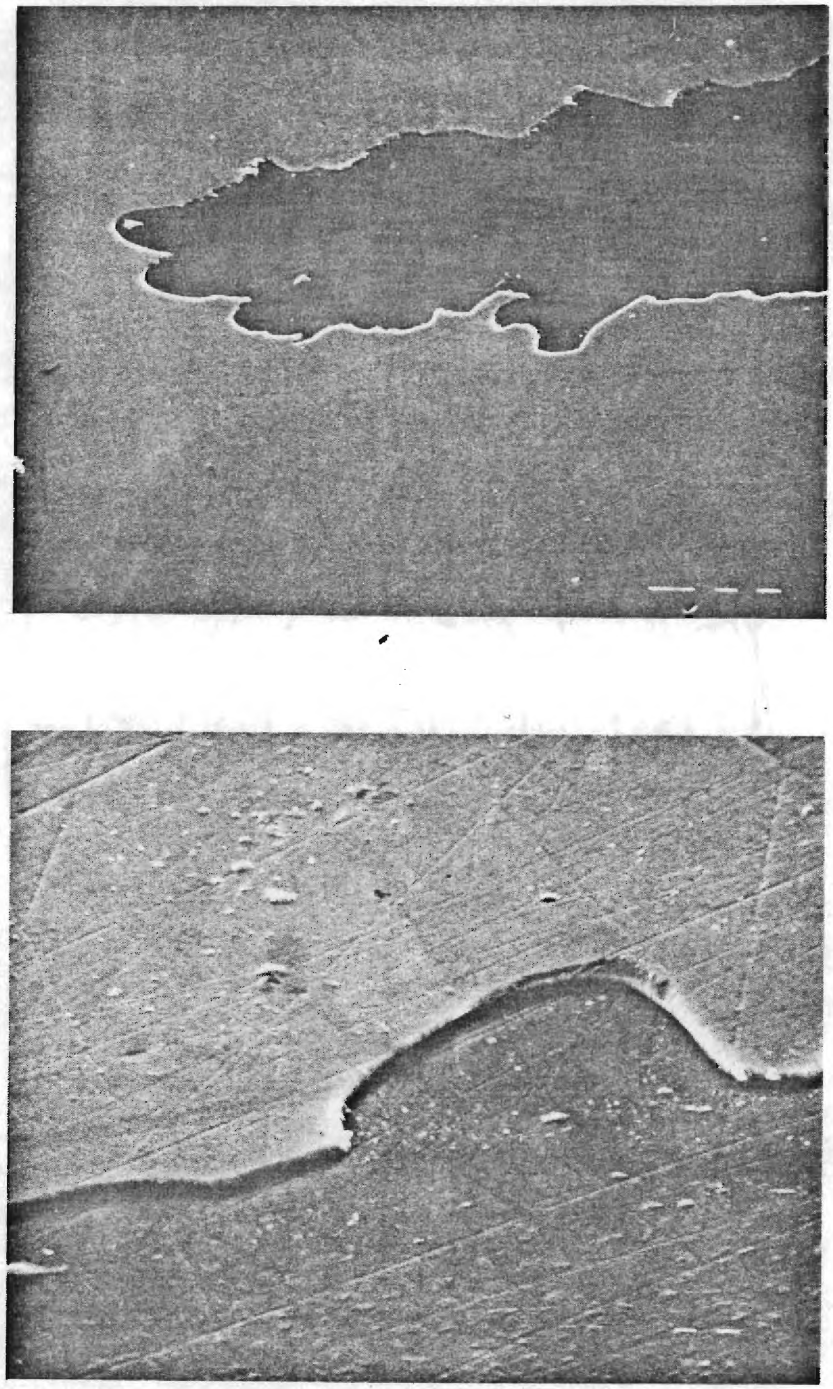


A



B

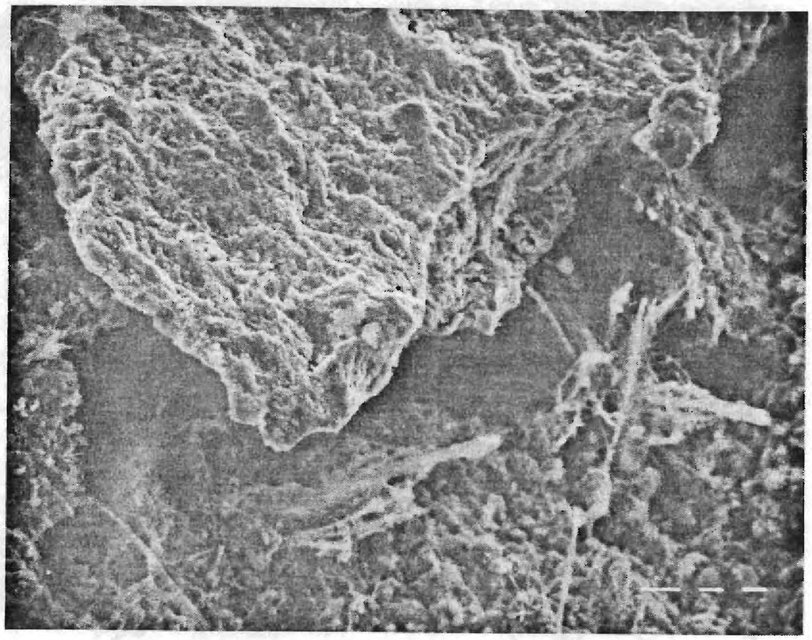
Figure 4. SEM micrograph of TiN-coated test sample after a 30 minute sliding friction test. (A) at 1600 X and (B) at 480 X show minor separation of TiN film at pitting artifacts and a wear groove produced by loose debris.



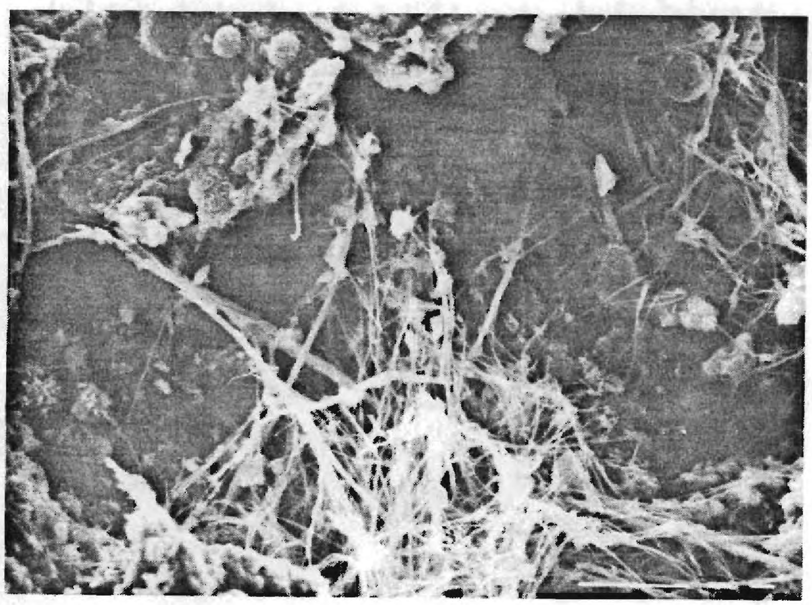
A

B

Figure 5. Scanning electron micrograph of sputtered Westinghouse material at (A) 600 X and at (B) 2800 X. Nodule-free, superficially homogeneous films are produced. An early spalled film edge is shown.

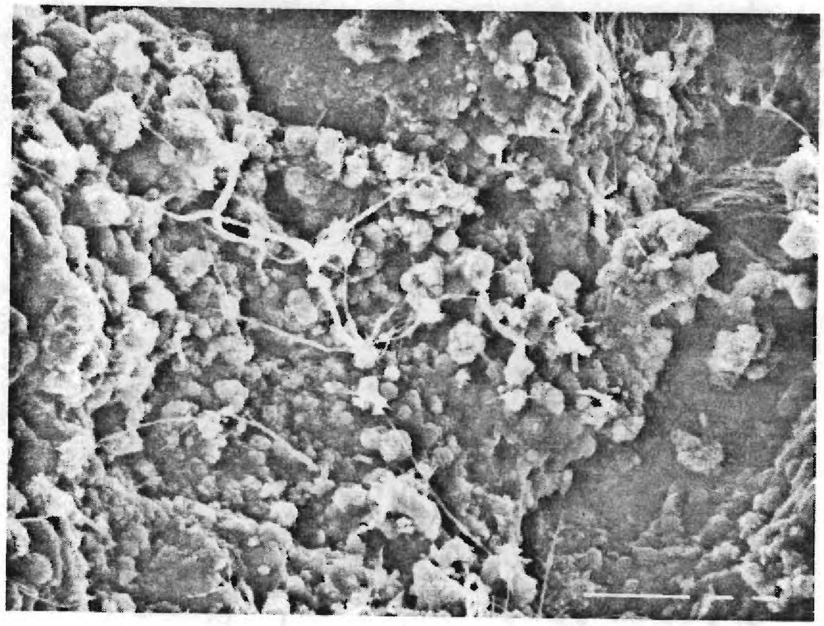


A



B

Figure 6. Fracture surface of the Westinghouse compact at (A) 510 X and at (B) 1460 X. Fibril and spheroid features representing different phases are noted in parent material.



A

B

Figure 7. Fracture surface of Westinghouse compact at (A) 1390 X and at (B) 2400 X. Spheroidal, fibril and platelet features representing several phases in the parent material are seen.

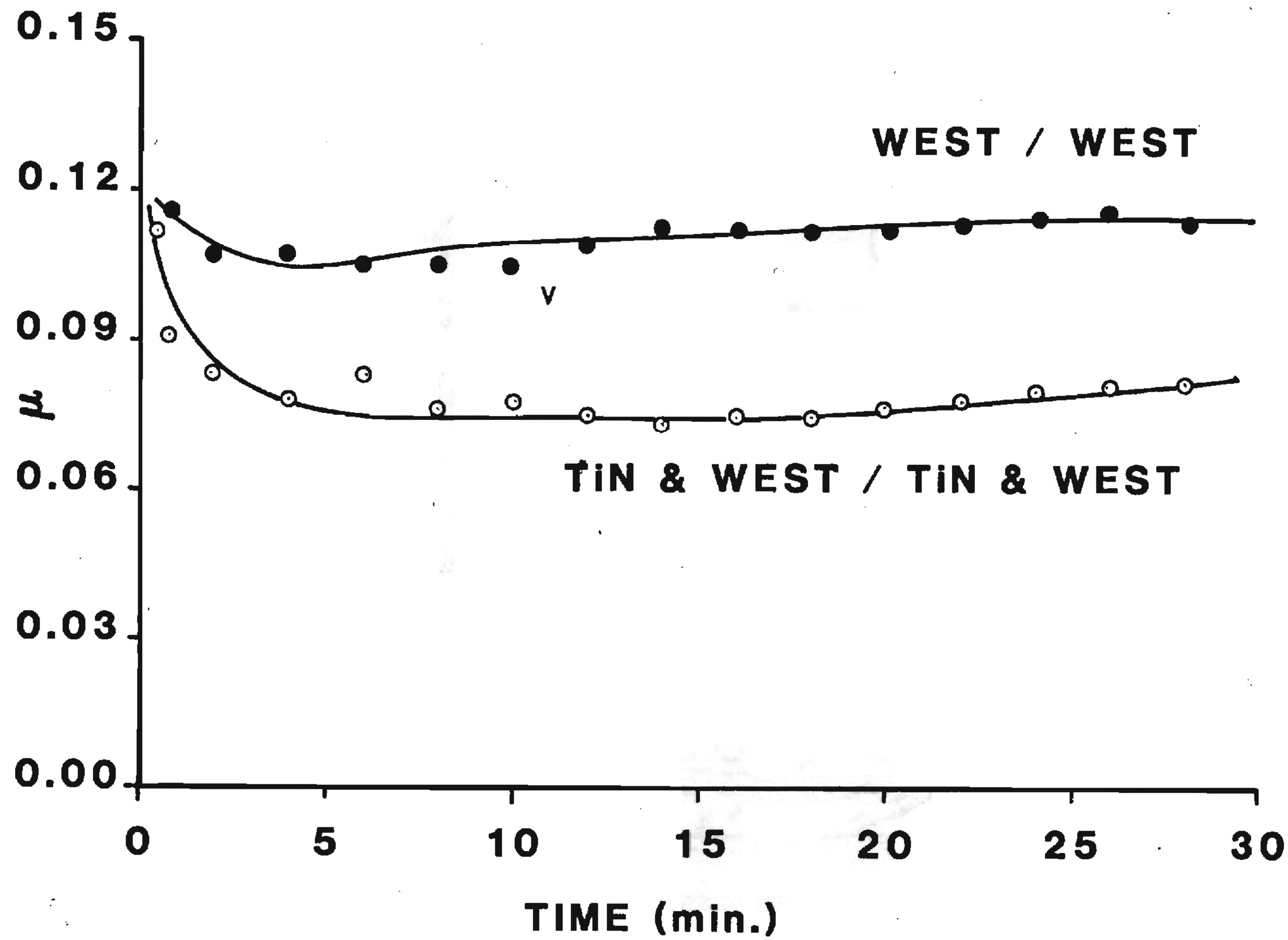


Figure 8. Measured coefficient of friction as a function of time in disc-to-disc sliding friction test. Data obtained with solid lubricant coated LFW-6 discs (Westinghouse material alone or duplex coated) are presented.

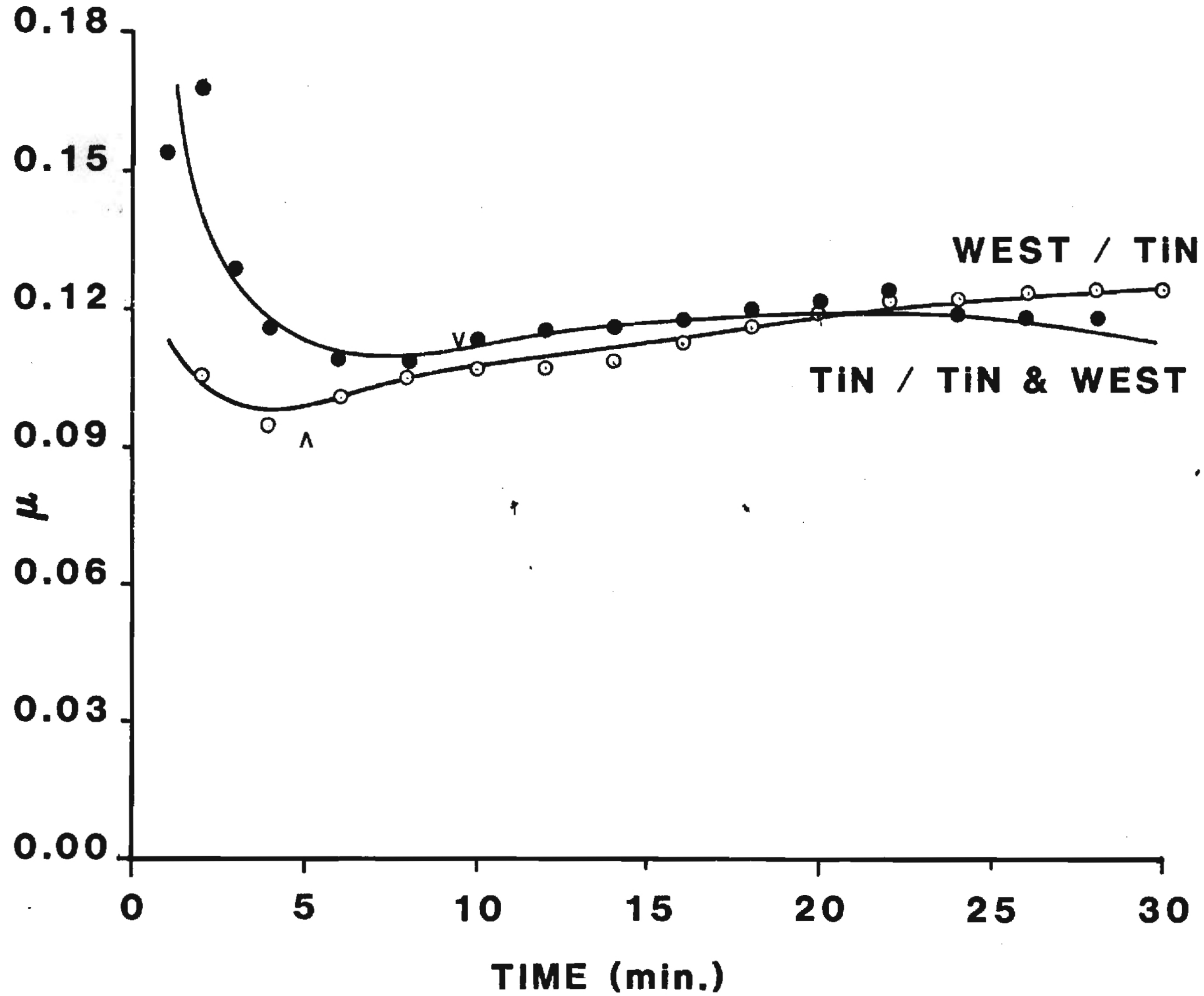
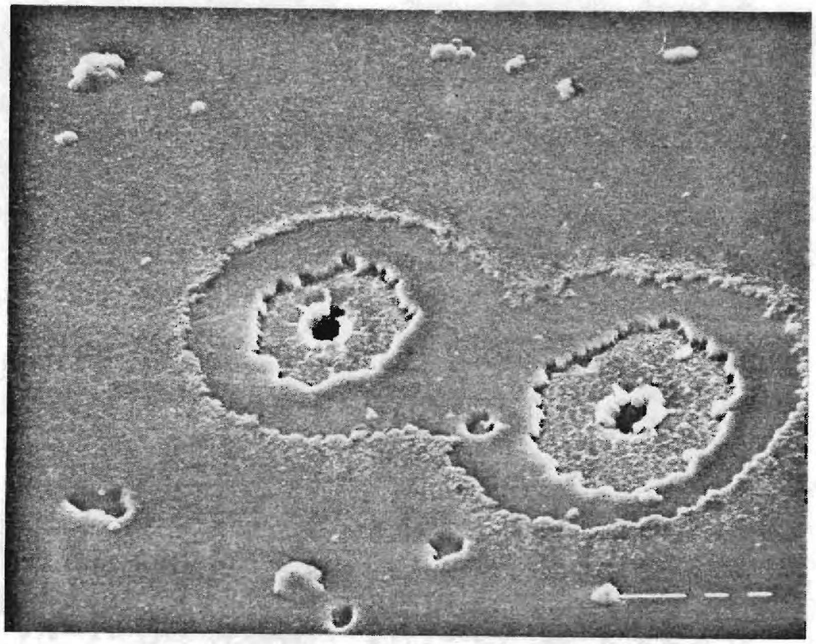


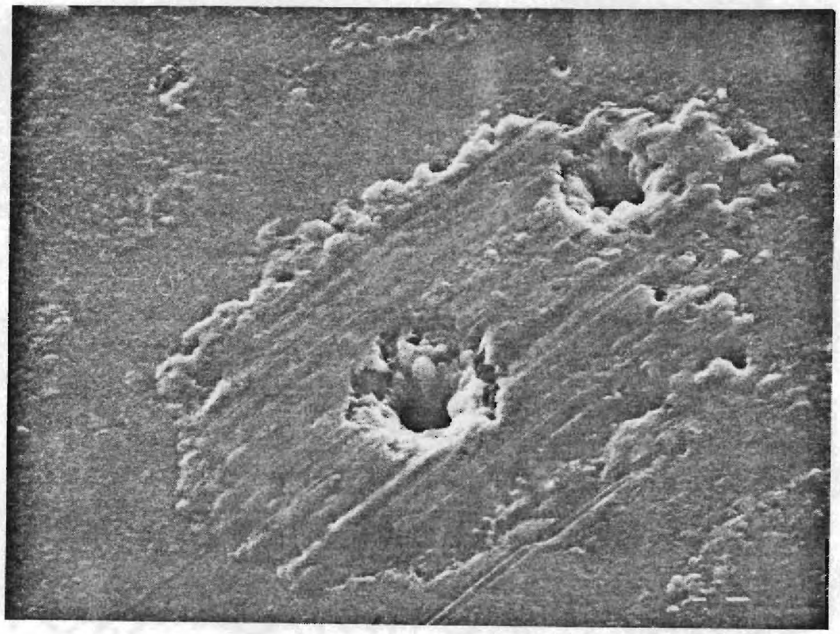
Figure 9. Measured coefficient of friction as a function of time in disc-to-disc sliding friction test. Data for solid lubricant-on-TiN and TiN-on-duplex coating.



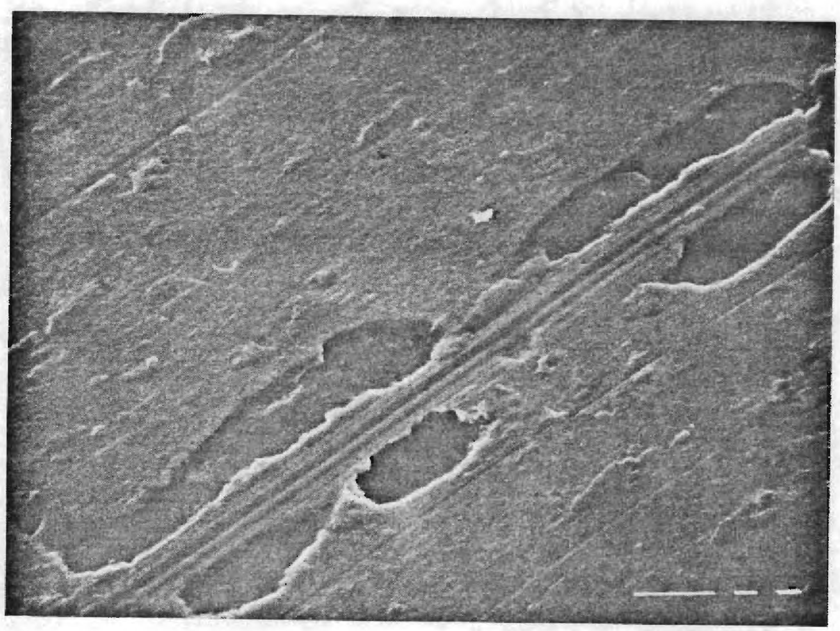
A

B

Figure 10. Duplex-coated, disc-to-disc LFW-6 test sample (A) before and (B) after sliding friction test at 1000 X and 1200 X respectively. There is little film loss at pitting artifacts.



C



D

Figure 10'. Duplex-coated, disc-to-disc test sample (C) showing pit action as a reservoir for the solid lubricant, and (D) localized film loss at isolated wear tracks. (C) at 1800 X. (D) at 1050 X.

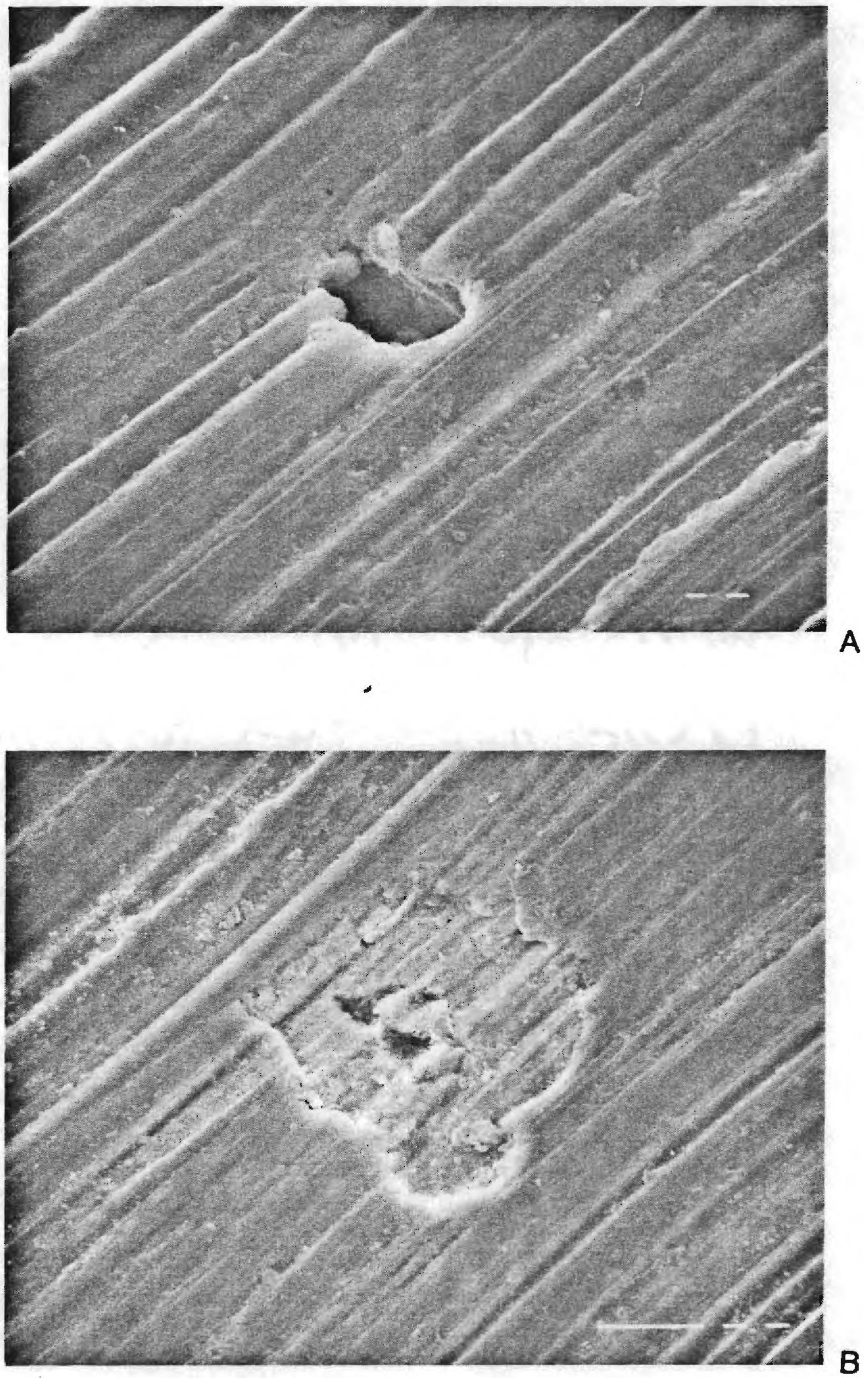
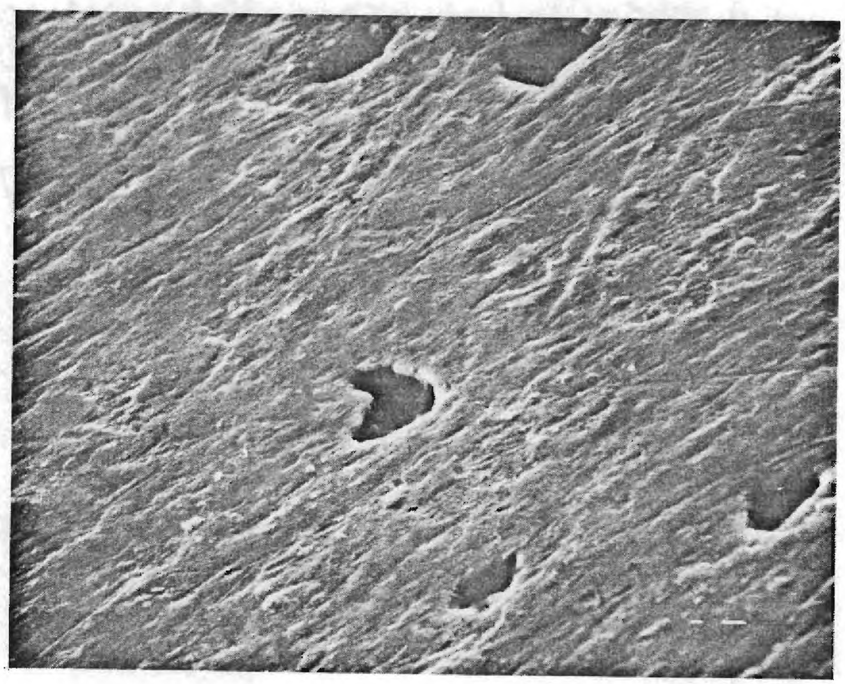


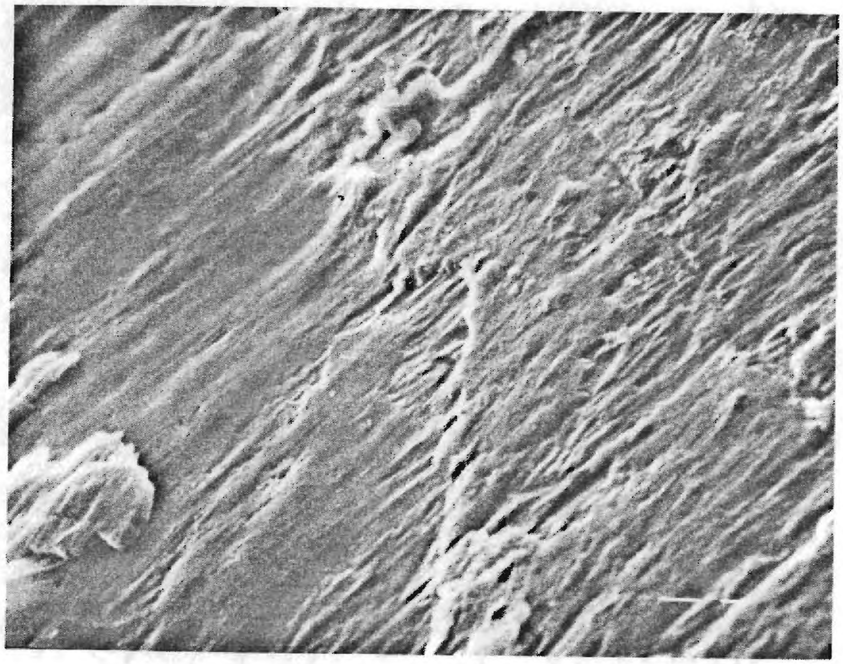
Figure 11. View of the TiN-coated ball track in the thrust bearing at 2600 X. Topography before rolling contact test is shown in (A). A similar location is shown at (B) at 1400 X following the rolling contact test. There is little film loss even at pitting artifacts.

A

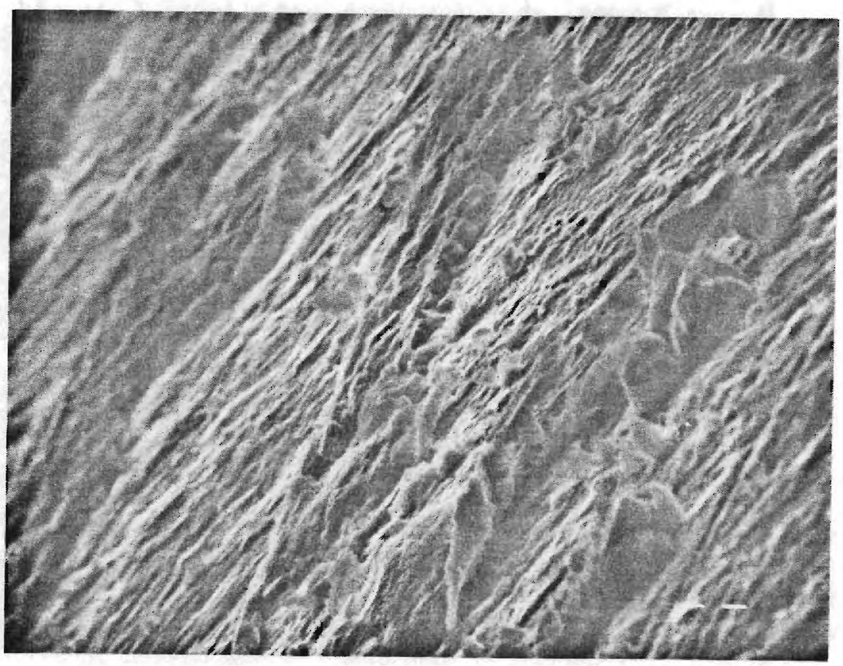


B

Figure 12. View of the ball track in (A) pure rolling region, and (B) in region with significant sliding. Micrographs at 2000 X and 1600 X respectively.



A



B

Figure 13. Ball topography following 30 minutes of testing. Transfer films of Delrin and TiN are produced. Micrographs (A) and (B) at 5500 X and at 2700 X.

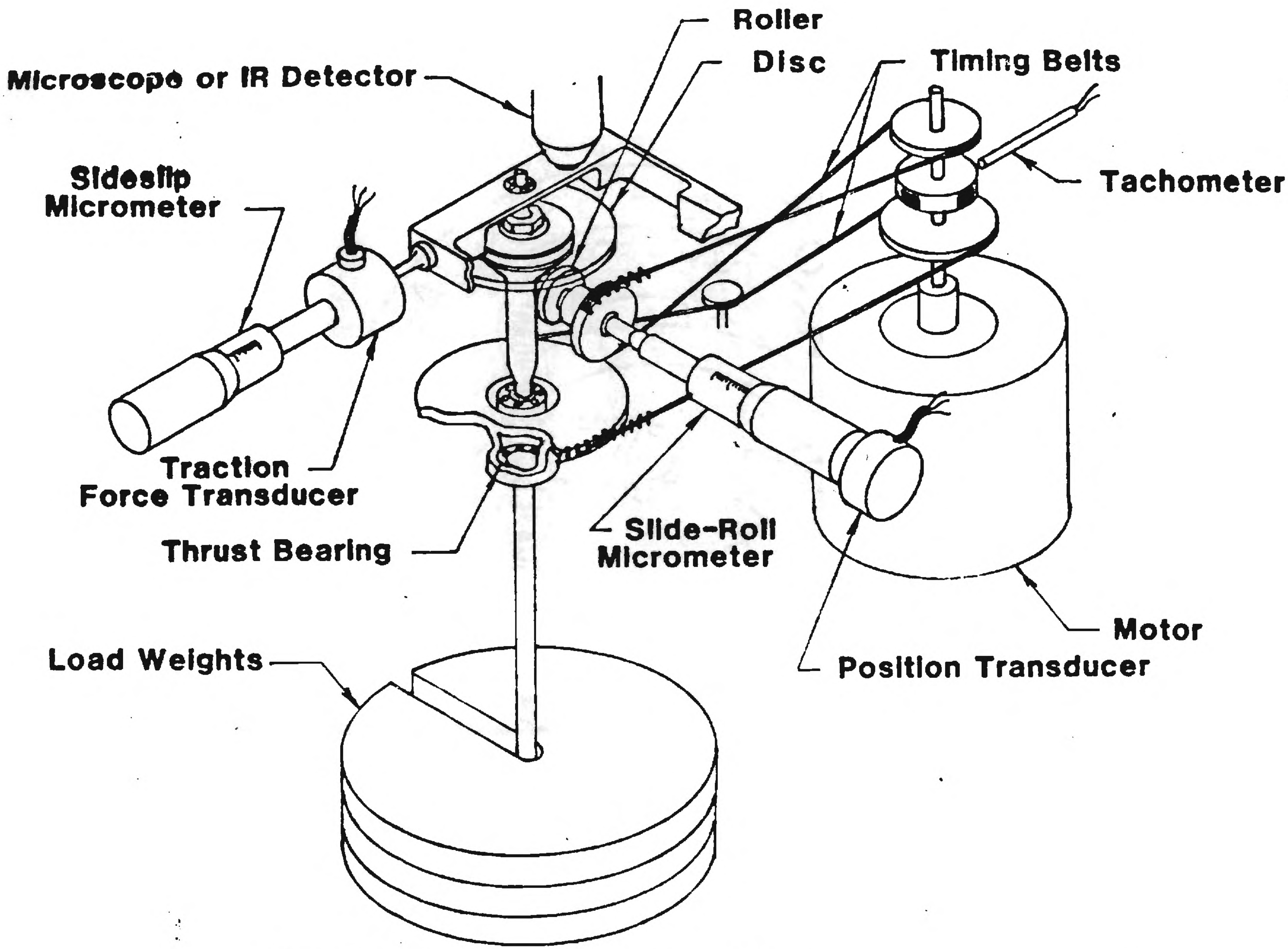


Figure 14. New concentrated contact simulator

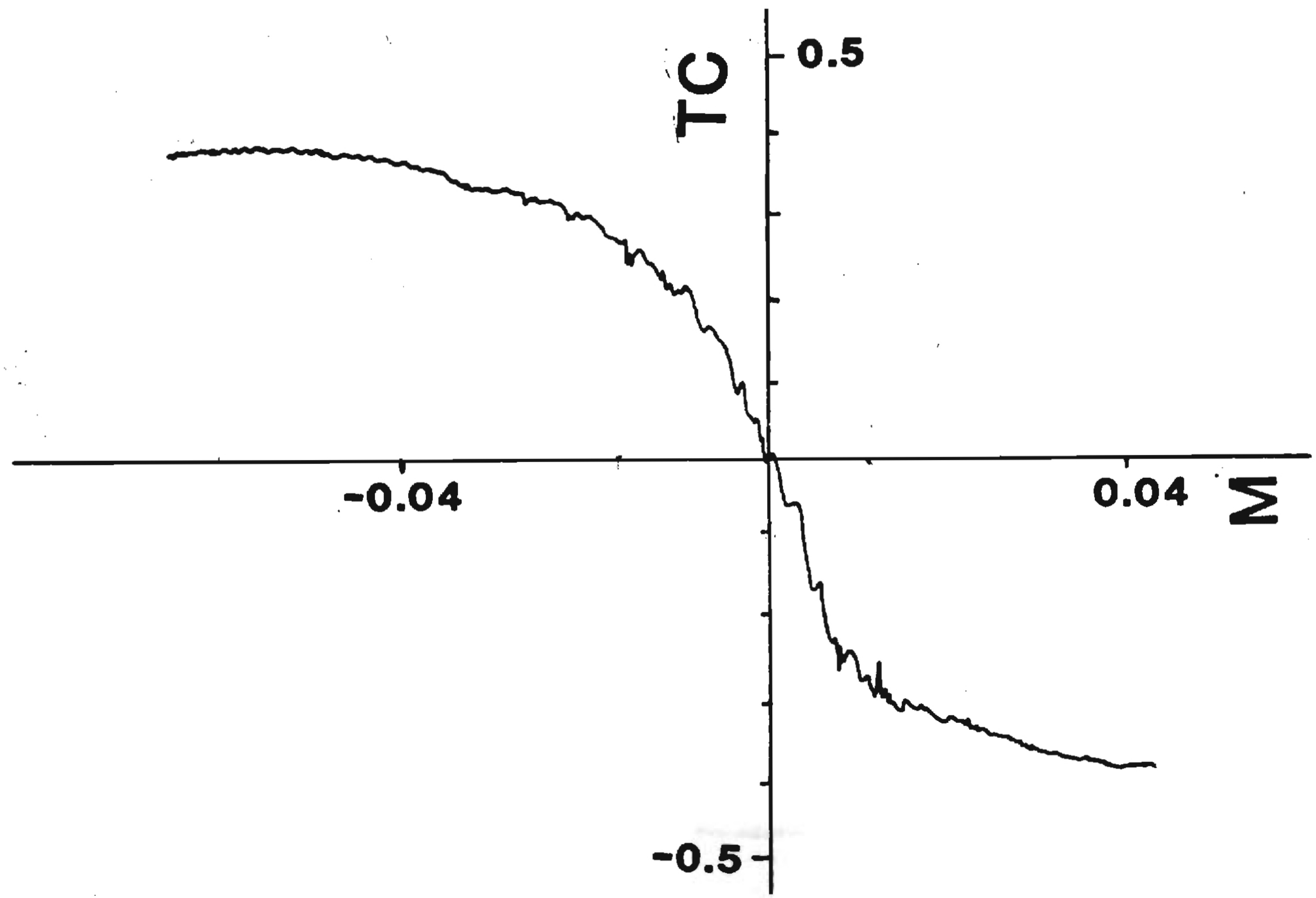


Figure 15. Uncoated 52100 steel at a Hertz pressure of 1.07 GPa temperature of 25C and rolling speed of 2 m/s after 10,000 revolutions of the roller.

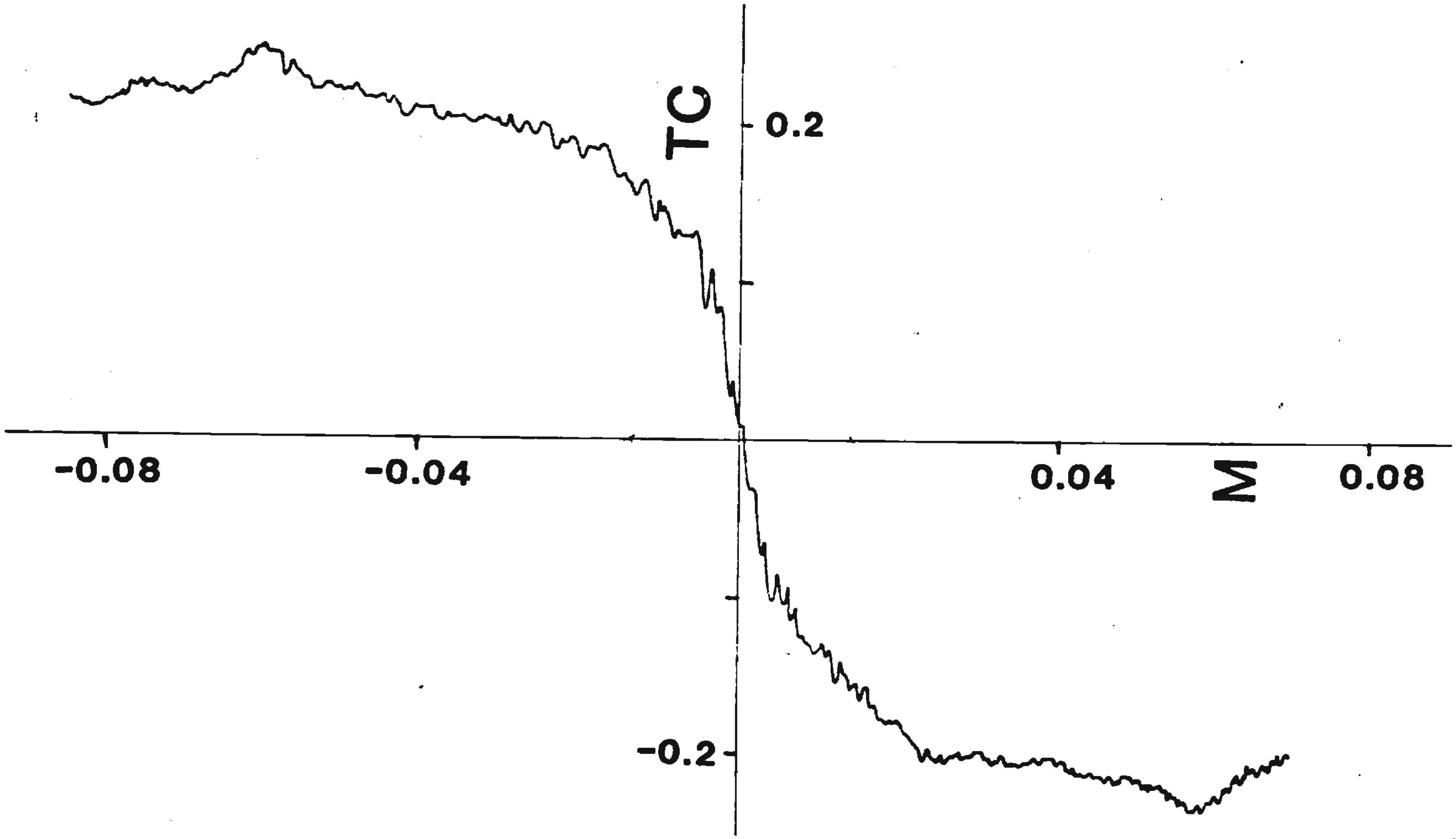


Figure 16. 0.4 µm coating of Ga/In/WSe on both 52100 elements at a Hertz pressure of 0.79 GPa, temperature of 23C and rolling speed of 2 m/s after 1500 revolutions of the roller.

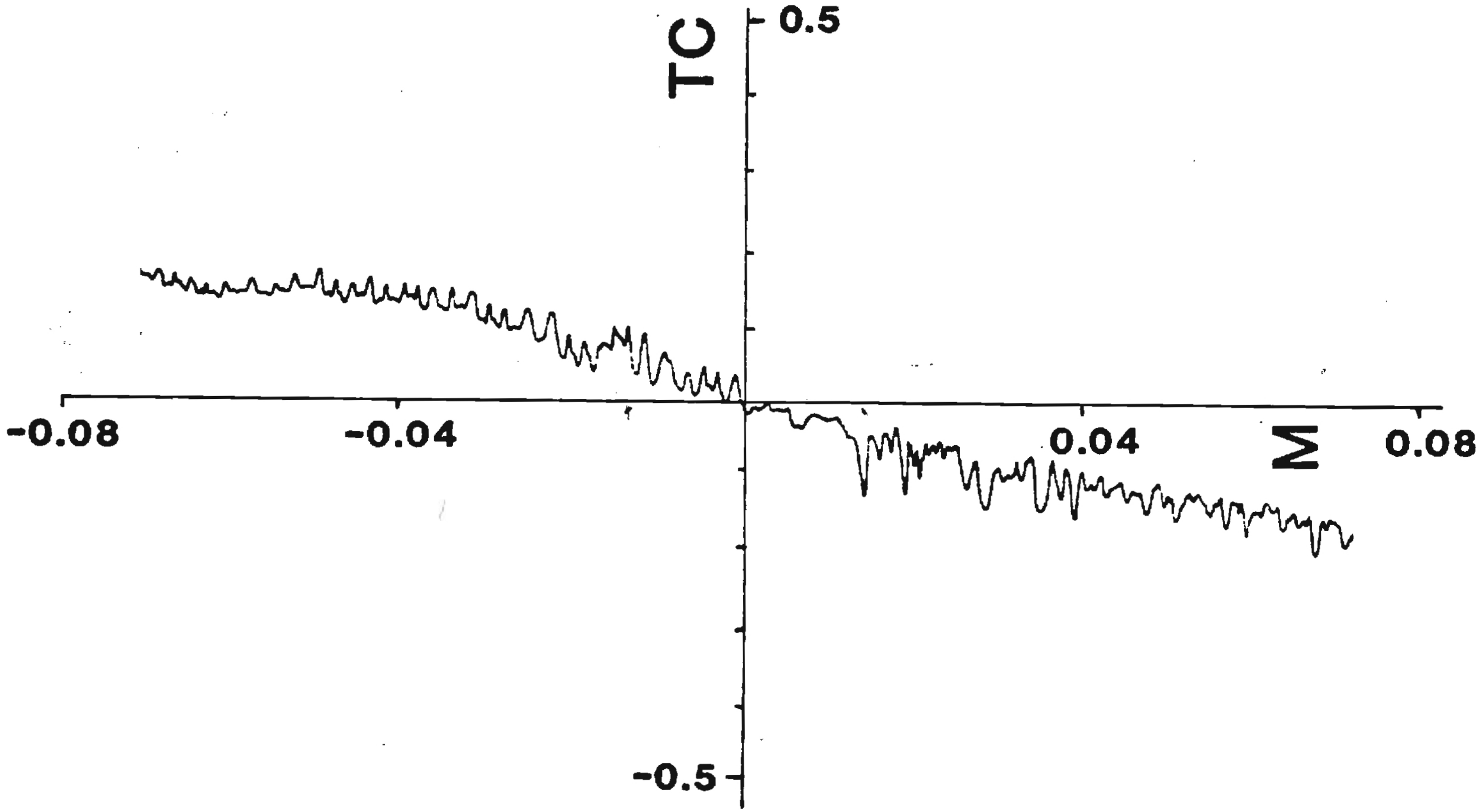


Figure 17. 0.4 μm coating of Ga/In/WSe on both 52100 elements at a Hertz pressure of 1.07 GPa, temperature of 26C, and rolling speed of 2 m/s after 2800 revolutions of the roller.

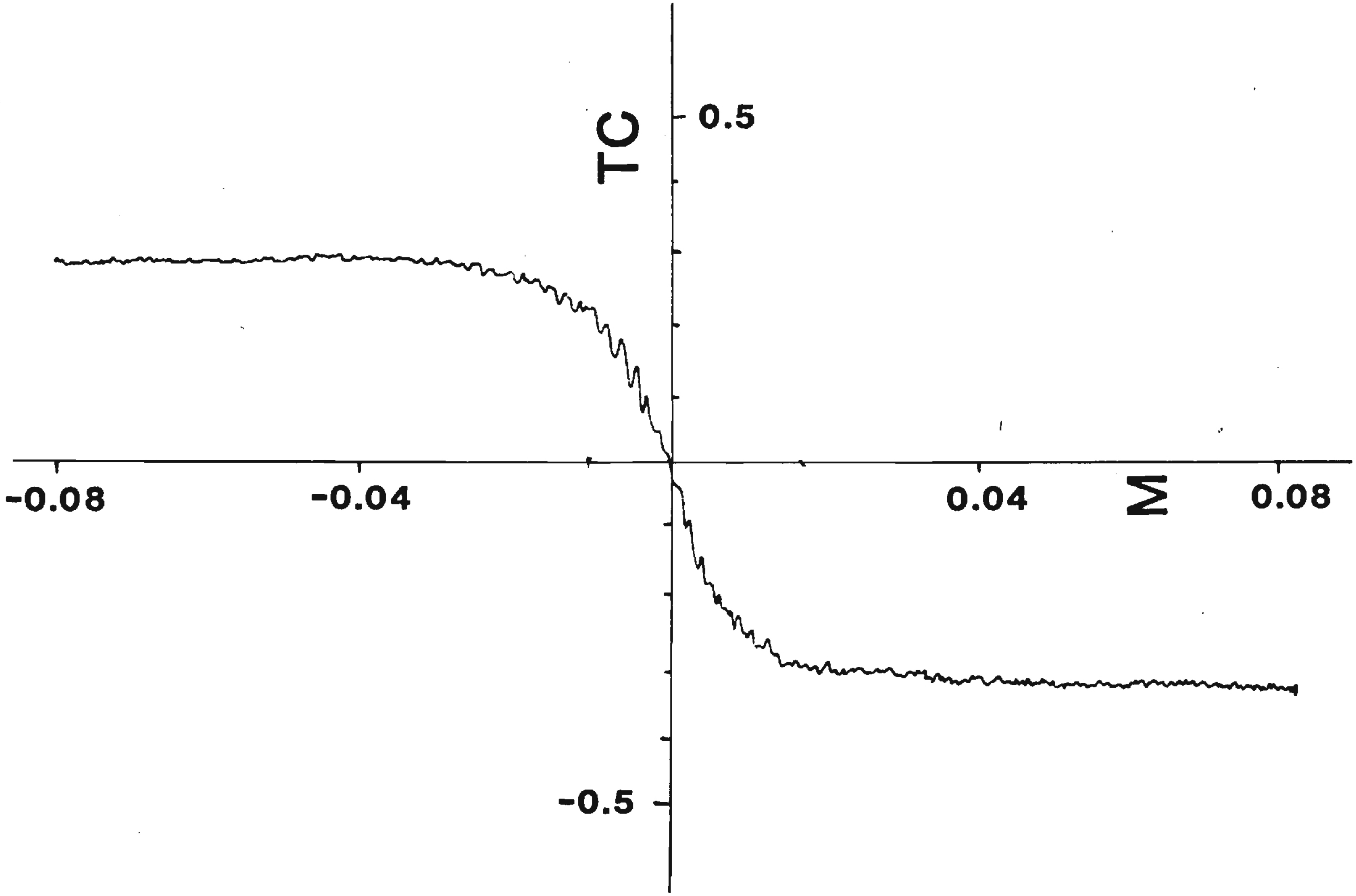


Figure 18. 0.4 μm coating of MoS_2 on both 52100 elements at a Hertz pressure of 1.07 GPa, temperature of 23C, and rolling speed of 2 m/s after 2,600 revolutions of the roller.

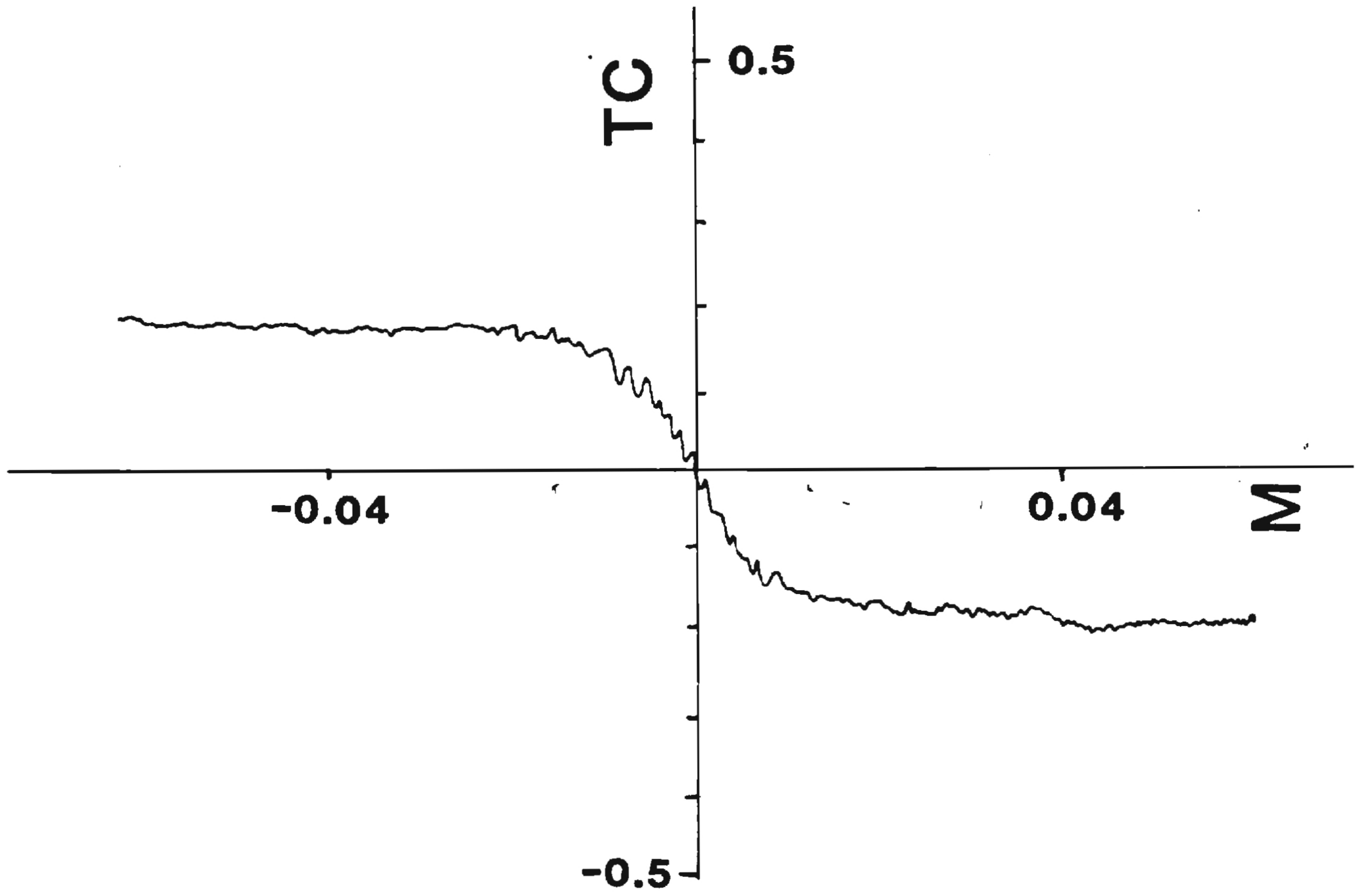


Figure 19. 1.5 µm coating of MoS_2 on both 52100 elements at a Hertz pressure of 1.07 GPa, temperature of 23C, and rolling speed of 2 m/s after 1800 revolutions of the roller.

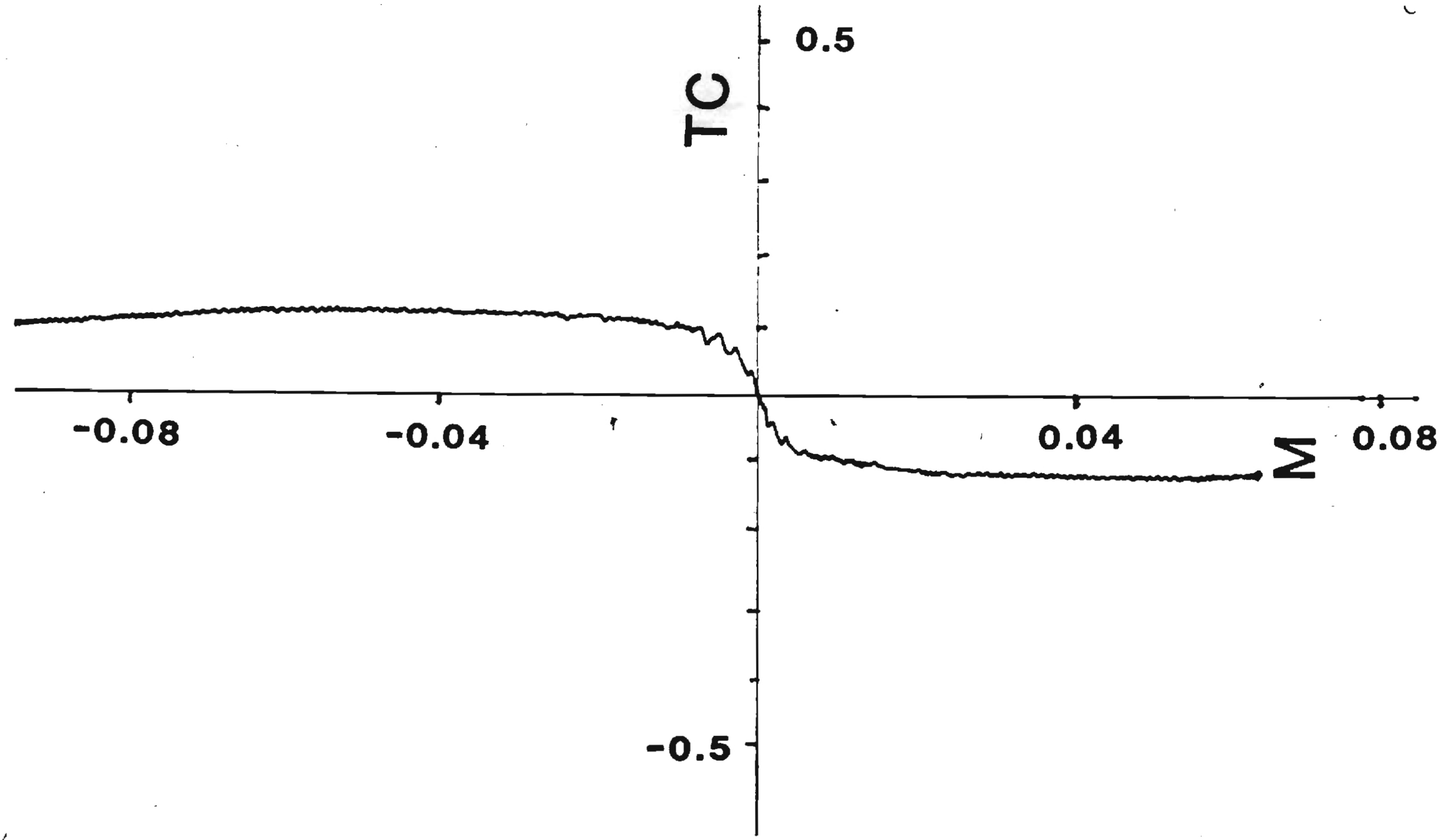


Figure 20. Burnished WSe_2 on both 52100 elements at a Hertz pressure of 1.07 GPa, temperature of 23C and rolling speed of 2 m/s after 3000 revolutions of the roller.

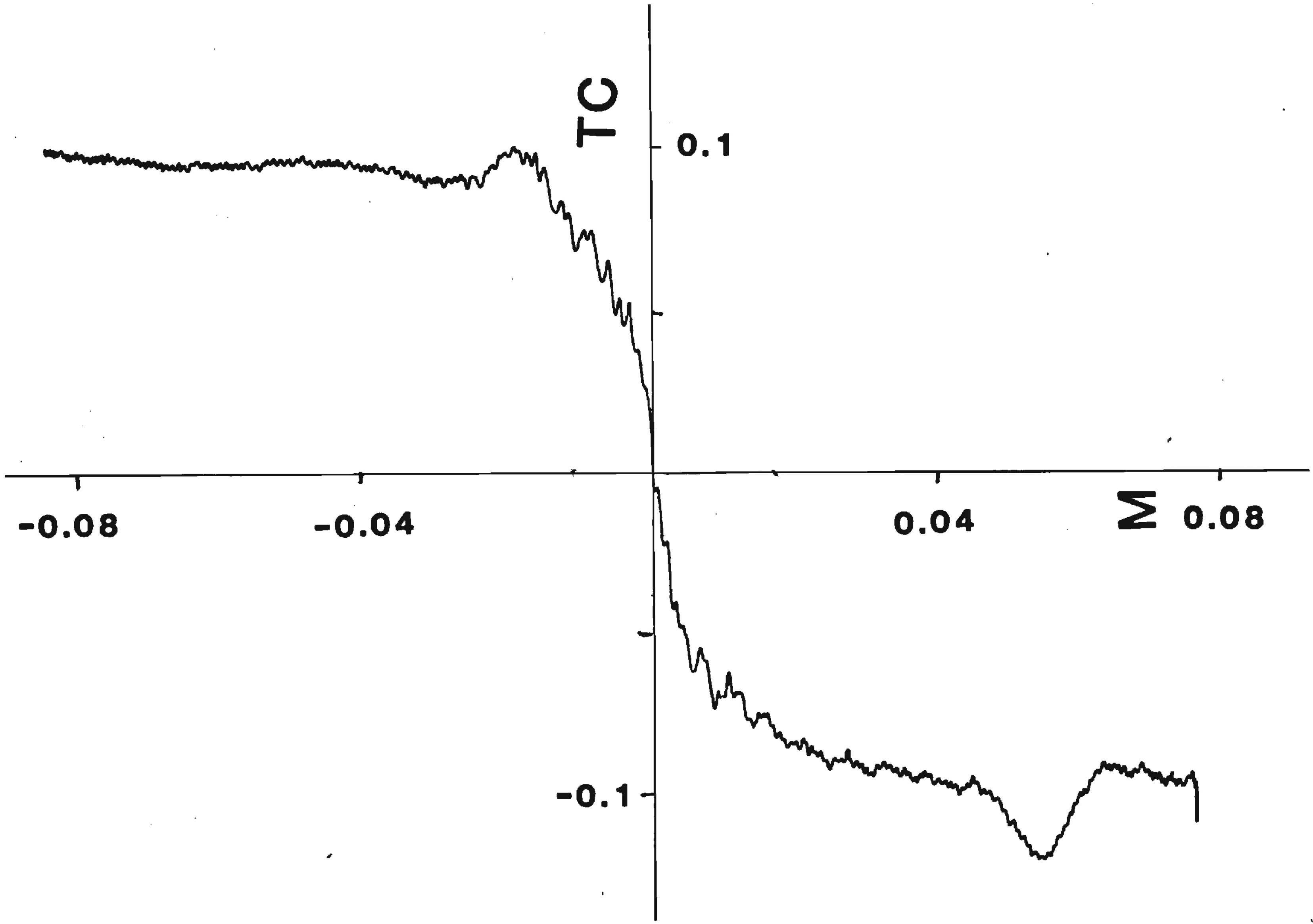


Figure 21. Burnished MoS_2 on both 52100 elements at a Hertz pressure of 1.07 GPa, temperature of 23C and rolling speed of 2 m/s after 1900 revolutions of the roller.

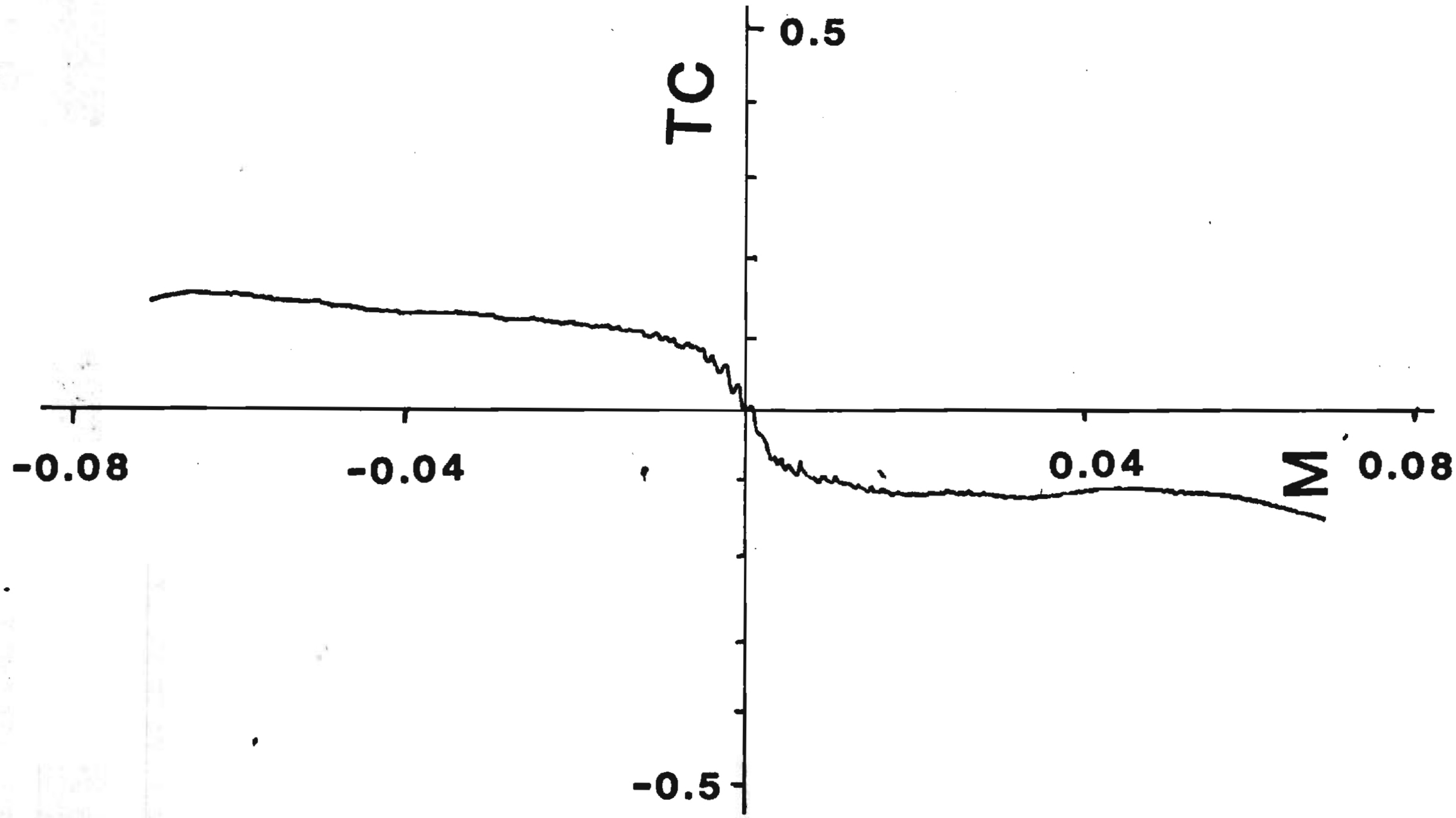
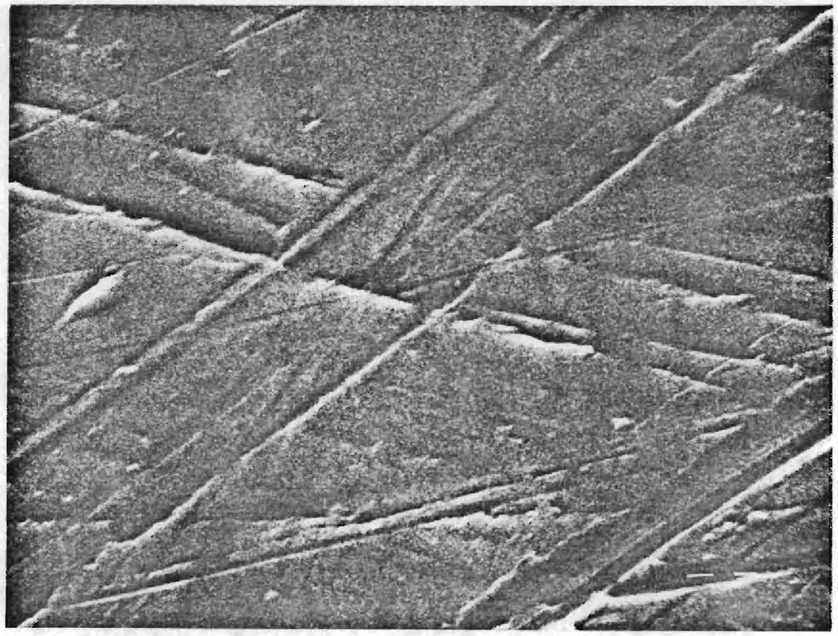


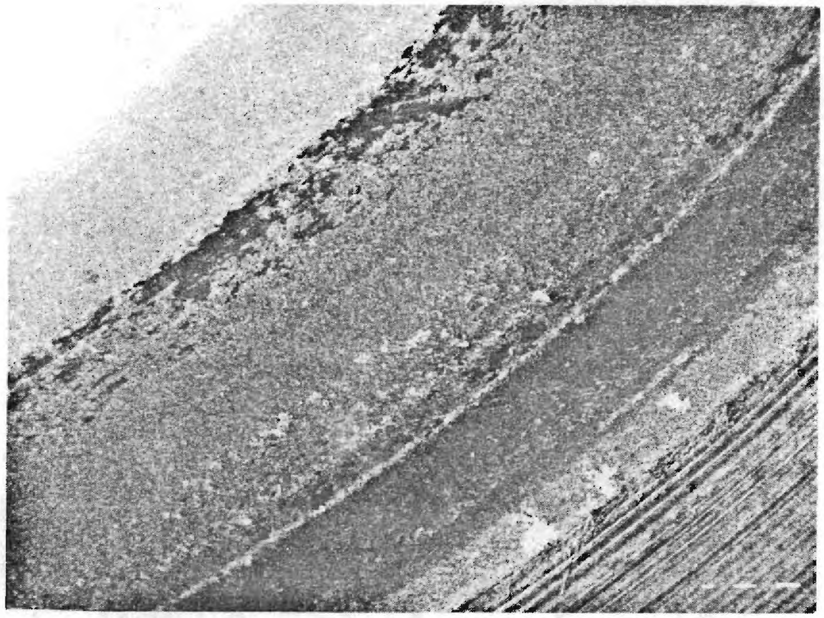
Figure 22. Burnished WSe_2 over sputtered TiN at a Hertz pressure of 1.07 GPa, temperature of 23C, and rolling speed of 2 m/s after 2300 revolutions of the roller.



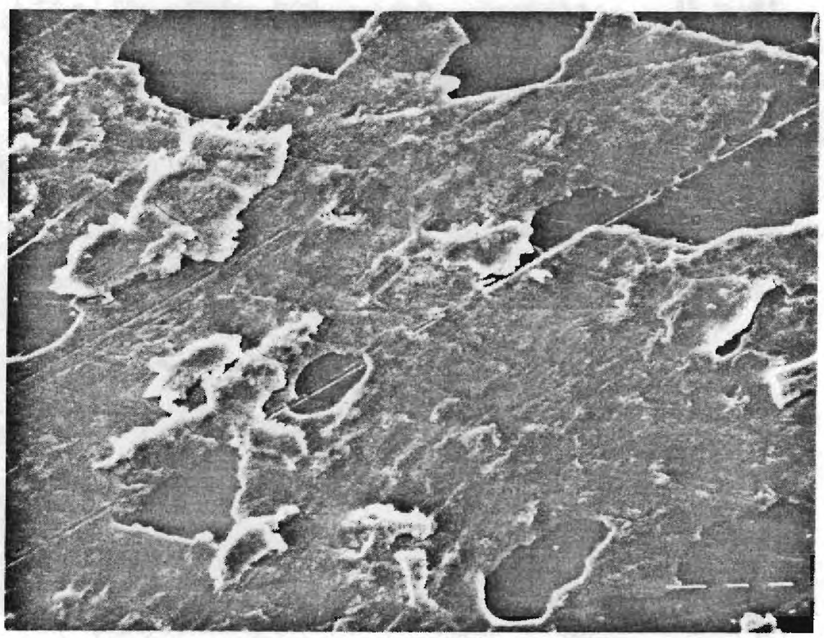
A

B

Figure 23. Topography of tungsten selenide coated traction disc (A) before and (B) after rolling contact tests in the concentrated contact test simulator. Magnifications are (A) 3600 X and (B) 520 X.

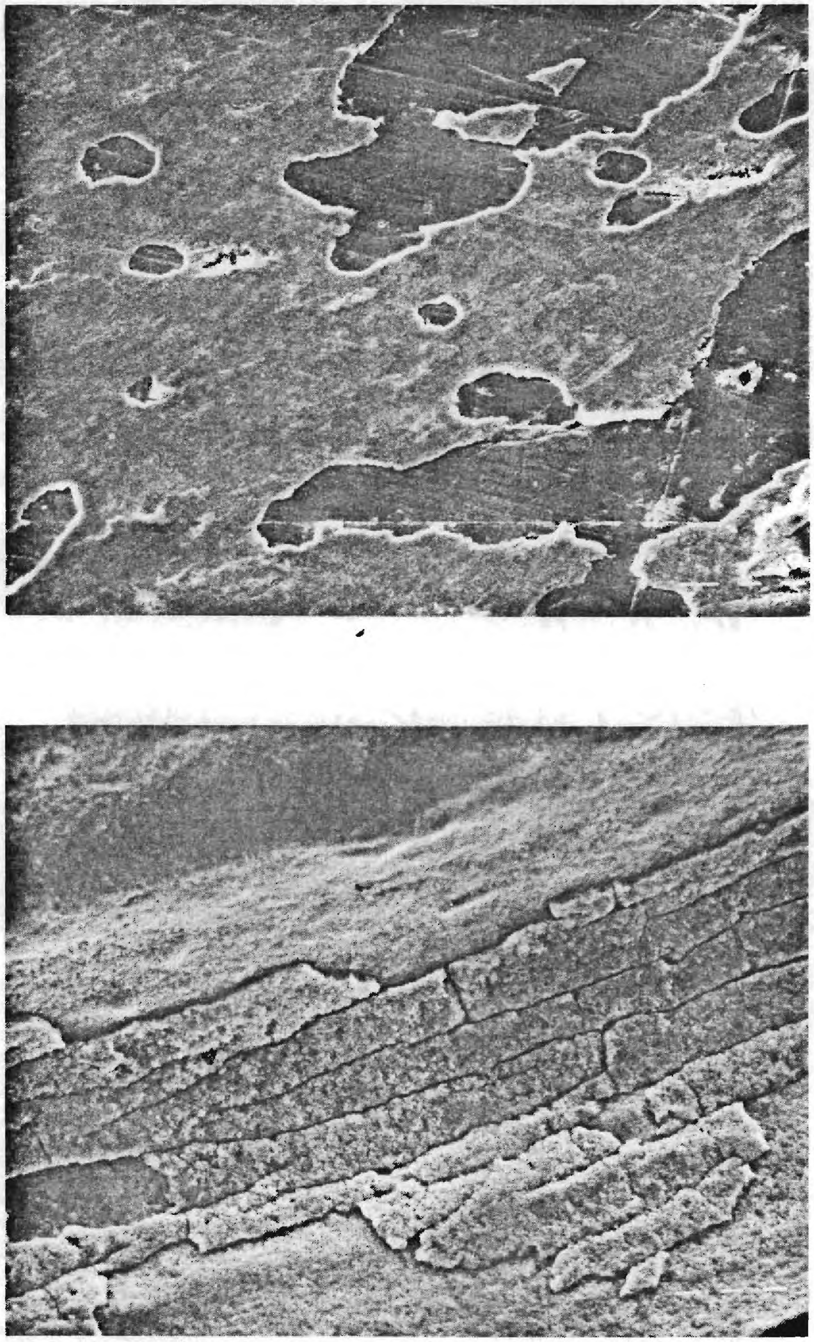


A



B

Figure 24. Low magnification view of the traction disc following the traction test. Debonded regions shown at 12 X in (A) are regions where there was approximately 4% sliding. Micrograph (B) at 520 X shows the debonded region at the inner edge of the traction disc.



A

B

Figure 25. Micrograph (A) shows the debonded region of traction disc outer edge at 520 X. Micrograph (B) illustrates the structure of burnished tungsten selenide film in a TiN-coated traction roller after the traction test.